Development and validation of a pilot juice extractor technique to process green sugarcane with brown leaf

Simiksha Balkissoon

Submitted in fulfilment of the requirements for the degree Master of Engineering in the Department of Chemical Engineering, Faculty of Engineering and the Built Environment at Durban University of Technology

Supervisor: Professor Sudesh Rathilal

Submission date: 12 March 2020

Declaration

I, Simiksha Balkissoon, declare that

(i) The research reported in this thesis, except where otherwise indicated, is my original work.

(ii) This thesis has not been submitted for any degree or examination at any other university.

(iii) This thesis does not contain other persons' data, pictures, graphs or other information, unless specifically acknowledged as being sourced from other persons.

(iv) This thesis does not contain other persons' writing, unless specifically acknowledged as being sourced from other researchers. Where other written sources have been quoted, then:

a) their words have been re-written, but the general information attributed to them has been referenced;

b) where their exact words have been used, their writing has been placed inside quotation marks, and referenced.

(v) This thesis does not contain text, graphics or tables copied and pasted from the Internet, unless specifically acknowledged, and the source being detailed in the thesis and reference section.

Student: Simiksha Balkissoon Signature: 

Supervisor: Professor Sudesh Rathilal Signature:

Acknowledgements

I would like to thank the following personnel at Gledhow Sugar Company as well as the Cane Testing Services at the Gledhow mill for their support, assistance and for the use of the mill for the validation of the pilot extractor; Steve Markham (Factory personnel), Queen Msane (Factory personnel), Arvin Maharaj (CTS lab personnel) and Zimkita Luvalo (CTS lab personnel).

I would also like to thank the following individuals for each of their specific roles played:

Professor Sudesh Rathilal for his committed support and assistance as a supervisor.

Sarah Weyer for her committed support and assistance.

Bryan Barker for his expertise in the field and assistance during the project.

Wanda Msani (SMRI heavy-duty laboratory technician) for his contribution to the experimental methodology.

Dr Richard Loubser for his assistance with the design and setup of the pilot extractor at the mill.

Dr Katherine Foxon and Steve Davis for their assistance with the interpretation of the results.

The analytical laboratory staff at the Sugar Milling Research Institute (SMRI) for the analyses performed on the samples.

The Department of Science and Technology for the co-funding of the Green cane harvesting project: Part 1 and the STEP-Bio Steering Committee for enabling this work to be completed.

Lastly, I would like to thank my husband for his assistance and advise as well as my family and the SMRI as a whole for their ongoing support during the course of my studies.

RESEARCH OUTPUTS

Conference Paper

Balkissoon, S., Barker, B., Weyer, S., Loubser, R. and Davis, S. B. 2019. Development and validation of a pilot juice extractor. Paper presented at the *South African Sugar Cane Technologists" Association*. Durban ICC, 21-22 August 2019. Sugar Milling Research Institute, 20.

Technical reports

Balkissoon, S., Weyer, S. and Barker, B. 2019. *Commissioning of a pilot juice extractor*. Report no. 2300.Durban: Sugar Milling Research Institute.

Balkissoon, S., Weyer, S. and Barker, B. 2019. *Validation of a pilot juice extractor against a commercial diffuser*. Report no. 2301.Durban: Sugar Milling Research Institute.

TABLE OF CONTENTS

List of Tables

List of figures

GLOSSARY

Ash content: Solid residue determined gravimetrically after incineration in the presence of oxygen.

Bagasse: The final crushed sugarcane fibre remaining after milling. The sugarcane residue leaving mills after the extraction of juice.

Blocking factor: A factor used to create blocks. It is some variable that has an effect on an experimental outcome but is itself of no interest.

Brown leaf: Previously referred to as trash but excluding tops.

Brix: The measure of dissolved solids in sugar juice, liquor or syrup using a refractometer, otherwise referred to as refractometric dry solids. For solutions containing only sugar and water, Brix = % sugar by mass. A unit used to express the concentration of solids in aqueous sugar solutions.

Conductivity ash: Estimate of ash content by measurement of the conductivity of the solution.

Extraction: Proportion of sugar extracted from sugarcane in the extraction plant; equals mass of sugar in raw juice as a percentage of mass of sugar in sugarcane.

Extraneous matter: All sugarcane leaves and tops, mud, soil, roots, rocks, stones and tramp iron delivered with the sugarcane.

Fibre: The dry fibrous insoluble structure of the sugarcane plant in which juice is stored and through which plant food, dissolved in water, is distributed throughout the plant.

ICUMSA colour: The ICUMSA colour scale is used to measure the grade and quality of the sugar. The colour of sugar directly relates to the degree of refining – raw sugars being dark brown in colour while highly refined sugars are white in colour.

Imbibition: The process of adding imbibition water to the extraction plant to increase extraction sometimes incorrectly referred to as maceration (steeping sugarcane in juice). Water added is called imbibition water.

Inversion: The conversion of sucrose in syrup into a mixture of equal amounts of glucose and fructose. The action is one of hydrolysis and may be carried out by the action of the enzyme invertase, or by heating with dilute acids. The liquid product from this process is called invert sugar.

Lixiviation: Process of separating soluble substances from insoluble substances by dissolving the former in water or some other solvent.

Preparation index (PI): The percentage of brix in the ruptured cells to total brix in the sugarcane after it has passed through sugarcane preparation equipment e.g. a shredder.

Pol: An approximate measure of the sucrose content of the sugar. Polarisation is measured by preparing a standard solution from the sugar and measuring the optical rotation of polarised light passing through a cell containing the solution. Sugar of 98 degrees' pol would contain about 98% sucrose.

Purity: The true purity is the sucrose content as a percentage of the dry substance or dissolved solids content. The solids consist of sugar plus non-sucrose components such as invert, ash, and colorants. Apparent purity is expressed as polarization divided by refractometer brix, multiplied by 100.

Reducing sugars: Reducing sugars are those which have the ability to chemically reduce (withdraw oxygen) from certain other chemical compounds. In milling and refining, reducing sugars (mainly glucose and fructose) are regarded as impurities.

Sucrose: sugar obtained from sugarcane: A white water-soluble crystalline carbohydrate, found naturally in many plants and extracted from sugarcane and sugar beets to make common sugar. Formula: $C_{12}H_{22}O_{11.}$ *Also called* saccharose.

Abstract

Annually, approximately 90% of the sugarcane planted in South Africa is burnt prior to harvest. The burning of sugarcane is a pre-harvesting technique that is well known to the sugar industry and one that has proven to be efficient. Due to the numerous associated disadvantages that exist, such as the public nuisance of soot and smoke, soil damage and its contributions to air pollution, it is foreseen that government legislation will become more stringent, prohibiting the burning of sugarcane, thereby forcing the industry to consider other alternatives. Processing green sugarcane with brown leaf is one of the sustainable alternatives to be considered which would introduce a beneficial option for both the sugar industry and the environment. For this alternative to be recommended as a viable option, an investigation was required to determine the effects of processing green sugarcane with brown leaf in a sugar factory specifically in alignment with their current juice extraction systems (diffusers).

Conducting such experiments on a commercial diffuser presented several challenges. A novel approach was undertaken to develop a pilot juice extraction technique to access and quantify the effects of processing green sugarcane with varying quantities of brown leaf in a sugarcane diffuser on a more controlled scale. Efforts were made to simulate conditions in a diffuser and thereby produce a juice that would closely represent the quality of juice extracted from a sugarcane diffuser.

A pilot juice extractor technique was designed, fabricated and its performance verified before determining suitable operating conditions for further experimental work. Experimental juice extraction systems and the applicability of the system to the proposed work was evaluated. The outcome of an extensive review of the literature revealed that the pilot juice extractor design had to be based on an upward forced-flow, submerged column with a steam jacket and electrical heating option. The pilot extractor showed good ability in differentiating between juices extracted from burnt and green sugarcane with and without brown leaf. The extracted juice quality was assessed and compared on the key analytes present in the extracted juice such as gravity purity (sucrose/brix), colour, conductivity ash, reducing sugars (fructose and glucose) and non-sucrose content. An experimental design allowed for key operating conditions of time (30, 45 and 60 minutes) and temperature (75 °C,

80 °C and 85 °C) to be tested. Suitable operating conditions for the pilot juice extractor, which emanated from the experiments, included a temperature of 80 °C and a retention time of 30 minutes. In addition, the juice concentrations in the pilot extractor were found to be different (higher or lower) to the concentrations of analytes present in the juice extracted from two established methods, namely cold digestion and press methods, for most juice quality parameters.

The pilot juice extractor performance was subsequently validated against a South African commercial diffuser for 16 different consignments of sugarcane of different varieties and included both green and burnt sugarcane. The diffuser draft juice was compared to the juice obtained from the pilot extractor, cold digestion and press processes. Due to a lack of green sugarcane samples tested at the factory, the correlations between draft juice and the extractor were derived for burnt sugarcane only. The pilot extractor juice quality for burnt sugarcane compared more favourably, in terms of the concentrations of the analytes, with the draft juice quality rather than the quality of the juice extracted from the cold digestion and pressing methods.

The investigation of the effects of varying quantities of brown leaf on the quality of juice extracted and the effect on the sugarcane density and percolation rate was carried out using the pilot juice extractor. The tests considered included four different sugarcane varieties (N12, N16, N47 and N39) to obtain a good representation of the different types of sugarcane that is processed in sugar factories. Results showed that an increase in brown leaf content adds to colour, conductivity ash and non-sucrose content and reduces purity, sugarcane density and percolation rates across all tested varieties. The pilot juice extractor presents a suitable method that can be utilised in future studies to assess factory specific combinations of sugarcane varieties, type (burnt or green) and the effects of adding brown leaf in a diffuser, in an effort to understand any potential factory processing impacts. This will aid factories in preparation for how best to handle the situation should they be required to process green harvested sugarcane with brown leaf in the near future.

1. INTRODUCTION

1.1 Background

Due to a worldwide transition towards a green environment, a number of industries are employing more environmentally friendly improvements or new alternatives to their processes to eliminate their negative carbon footprint. The pressure imposed on the environment by the sugar industry stems from the well-known and widespread practise of burning sugarcane on the fields prior to harvest. Madho *et al.* (2017) reported that approximately 90 % of sugarcane was burnt prior to harvest in the 2016/17 season.

One of the primary reasons for the burning of sugarcane is to improve processing of the sugarcane due to removal of unwanted parts of the sugarcane plant, namely brown leaf (previously referred to as trash) which would have to otherwise be manually removed (Bernhardt and Pillay, 2000). Approximately 80% of the unwanted content is burned off. Inclusion of these components results in several implications in downstream processes such as extraction, diffusion, juice clarification and so forth, eventually leading to sugar losses and poor sugar quality. Other reasons for burning sugarcane include killing microorganisms, reduction in manual labour, transportation costs and killing of harmful bodies such as scorpions and snakes, which are harmful to labourers (Bernhardt and Pillay, 2000). However, the burning of sugarcane poses several disadvantages such as faster deterioration between burn to crush delays, poor soil conservation and, most importantly, air pollution (Bernhardt and Pillay, 2000).

It is foreseen that the sugar industry may face amended regulations and thus will be prohibited from burning sugarcane in the near future, due to the impact on the environment. Such a situation will force the industry to look towards alternative sustainable methods of harvesting sugarcane. Van Antwerpen (2010) mentions that de-leafing the sugarcane prior to delivery as opposed to burning serves as a desirable option for the reasons that it promises improved yields, reduced deterioration losses and soil and water conservation. Another desirable option includes co-processing the brown leaf with green sugarcane. However, any new method must consider the effect on supply chain operations, as well as the quality and

recovery of sugar with brown leaf in a sugar factory. In addition, the harvesting of green sugarcane affords the industry the opportunity to explore the value of the brown leaf fibres that would normally be lost to burning in the field. Several countries such as Brazil have already begun to use or explore the leaves as a source of fuel for electricity and a number of studies have been conducted in alignment with this (Van Antwerpen, 2010). Harvesting techniques in this study will refer to and differentiate between burnt sugarcane and green sugarcane (with brown leaf). The definition of trash varies vastly in literature. Trash may refer to leaves and possibly tops in previous studies whereas in this study it will be referred to as brown leaf (excludes tops).

The main process in a sugar factory associated with the incoming sugarcane is referred to as the juice extraction process, which occurs in a milling tandem (employed by approximately 14 % of the South African factories) and diffusers (employed by about 86% of the factories). The effect of harvested sugarcane (burnt or green with varying % brown leaf) must therefore be conducted in alignment with the abovementioned process units to assess whether brown leaf separation prior to processing or co-processing the brown leaf with the sugarcane would be more favourable. An understanding of the in-factory processing challenges and costs associated with the sugarcane harvested by each method will provide insight as to whether the alternative should be considered.

1.2 Rationale

For over 60 years, a substantial amount of research has been conducted and reported on milling tandems with respect to processing green sugarcane. Previous research (Bernhardt and Pillay, 2000; Muir *et al.*, 2009; Muir and Eggleston, 2009) investigated the effects of green sugarcane harvesting on diffusers using laboratory and full-scale processing trials; however, the results were generally inconsistent and deemed inconclusive for the purpose of quantifying the operational and cost impacts. This study presents work relating to processing green sugarcane with brown leaf in diffusers, for which information is lacking to this date but required since majority of the sugar factories now operate on diffusers as elucidated by current statistics. The lack of experimental work in this area is due to the logistics around carrying out such trials in a large commercial diffuser, which serves as a limitation (Barker, 2017a). The limitations are associated with the lack of control over the variation of sugarcane

entering the diffuser under normal operating conditions and the difficulty associated with varying quality characteristics of the extracted juice. The change in sugarcane consignments occurs more frequently than the average juice and fibre retention times. Such confounding conditions pose difficulties in obtaining meaningful and reliable results.

Previous work investigated the impact of brown leaf on juice and sugar quality involved cold digestion, by the direct analysis of cane (DAC) as an extraction method (Reid and Lionnet, 1989) and a press extraction method (Scott *et al*., 1978). Neither of these methods is representative of conditions that exist inside a diffuser. Cold digestion (Buchanan, 1967) is designed to open all cells by high-speed shearing of sugarcane fibre into pulp to release all dissolvable material and it is therefore believed that cold digestion extracts more brix and non-sucrose material than diffusion. The press method is different from diffusion as the press merely squeezes the juice out of opened cells and therefore will extract less brix and non-sucrose material than a diffuser.

1.3 Aim and objectives

The main objective of the study was to assess the impact of combinations of sugarcane varieties and brown leaf in a diffuser extraction process should factories be required to process green harvested sugarcane. It was therefore proposed to develop a technique for extracting juice that is representative of that from a commercial diffuser, since the idea of carrying out trials in a full-scale diffuser would be impractical. The abovementioned reasons sparked the necessity for a pilot juice extractor technique to be designed and developed to achieve this. The pilot juice extractor presented the opportunity to conduct more versatile, rapid and controlled experiments to be performed by circumventing the limitations associated with full-scale diffuser trials.

The specific objectives used in the study to achieve the main objective are grouped into three stages. Part one involved establishing if significant differences could be detected in the quality of juice extracted from green sugarcane with and without brown leaf using the pilot juice extractor. It also included defining suitable operating conditions of the pilot juice extractor, specifically with regards to temperature and time to be used for the validation phase later on and to finally compare the quality of the juice extracted from the pilot extractor to juice obtained from two established methods namely cold digestion and pressing. Part two

of the study aimed at establishing a method for extracting a juice with a quality representative of a commercial diffuser, validating the pilot extractor juice against the juice from a commercial diffuser for different sugarcane varieties and lastly comparing the pilot extractor juice, the Direct Analysis of Cane (DAC) extract and pressed juices to the draft juice. Part three focussed on investigating the effect of sugarcane variety, type and varying quantities of brown leaf on the quality of juice from a pilot juice extractor for different sugarcane varieties as well as on the shredded sugarcane density and percolation rate for different sugarcane varieties.

1.4 Structure of the dissertation

Chapter 2 in the study contains a detailed review of literature providing an overview of the nature and composition of sugarcane, the solid-liquid extraction process in the sugar industry, the sugarcane diffuser process and factors affecting the process, a review of suitable analytical methods and previous experimental work relating to the study. Chapter 3 outlines the experimental methodology followed for all three phases of the study. Chapter 4 includes the results and discussion presented with the use of tables and graphs. Finally, chapter 5 concludes all findings that emanated from the results of the study.

2. LITERATURE REVIEW

The objectives of the literature study were to:

- Understand the extraction mechanism in a diffuser and to apply it to the proposed work.
- To evaluate existing experimental juice extraction systems and the applicability of these systems to the proposed work.
- Identify key factors that affect extraction in a full-scale diffuser and to use the information for the design and development of a pilot juice extractor method.
- Recommend processing conditions to be used in the pilot juice extraction experiments for the proposed work by reviewing previous diffuser related experimental work.

2.1 Nature and composition of sugarcane and the effect on extraction

Studies manifest that the nature and composition of sugarcane plays a significant role in the extraction process. The impurities are said to originate from the sugarcane itself or can be formed during the process. The extraction of such impurities can result in the presence of such components downstream in the sugar process resulting in large processing costs. Several studies conducted with regards to the nature and composition of sugarcane, explaining exactly how it can affect the extraction process was reviewed to conclude important findings that applied to the scope of the proposed study.

2.1.1 Nature and composition

Understanding the basics of the structure of the sugarcane plant allows one to establish how the raw material will behave in an extraction process. Sugarcane is defined as a giant grass (Lionnet, 1985). The stalk (the section located above the root level to below the immature top) of the sugarcane consists of long internodes, soft centres and an external hard rind (protective skin). The stalk is typically cleaned before reaching the miller by a well-known harvesting technique, which involves burning the sugarcane in the fields. This technique

allows for the removal of most of the unwanted material, which is not part of the stalk, namely tops and brown leaf.

Figure 2-1 illustrates the different components of a whole sugarcane plant. Tops are described as the portion of stalk above the natural breaking point, namely the leaves and leaf sheaves that may be green or brown (dead) and attached to that part of the stalk. Eggleston *et al.* (2009) mentions that various authors define trash as leaves with or without green tops. Trash, as referred to by Lionnet (1985), is considered as the green or dead leaves and sheaves which lie below the natural breaking point of the stalk (Figure 2-1). This is now commonly referred to as brown leaf.

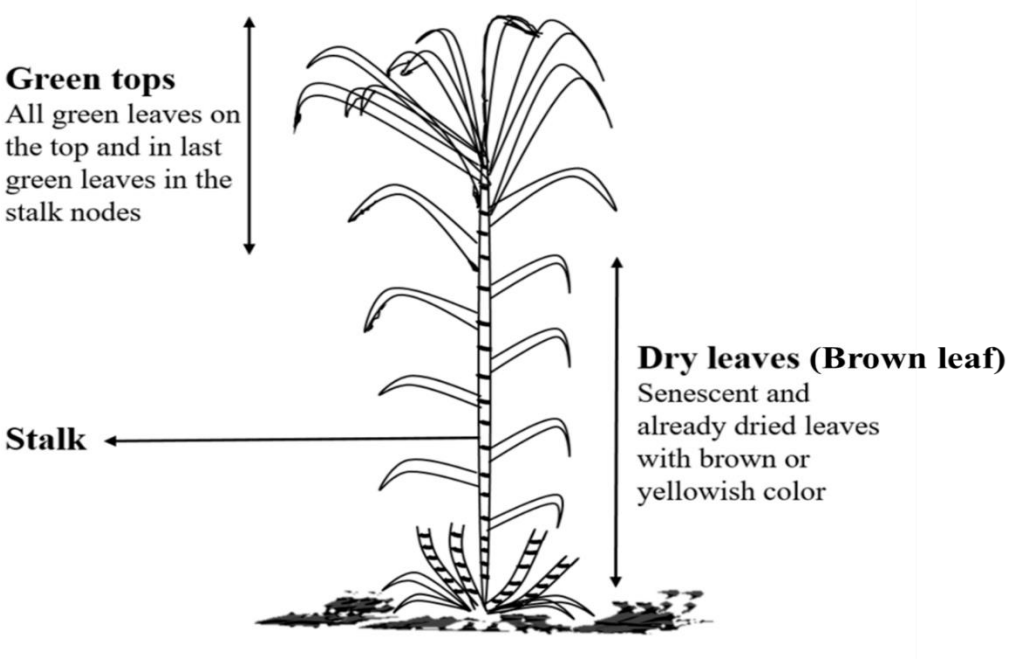

Figure 2-1: Reproduced schematic representation of sugarcane plant separating in green tops, dry leaves and stalk (Menandro, 2017)

There are two types of sucrose-bearing cells in the sugarcane stalk, parenchyma cells (soft-walled) and sclerenchyma cells (small elongated) as described by Rein (2007). Both types of cells contain sucrose, although most of the sucrose is found in the large soft-walled cells on the inside tissues while a smaller amount can be found in tough, small-elongated cells. A higher concentration of sucrose and a lower concentration of non-sucrose is found in the parenchyma cells compared to the sclerenchyma cells (Rein, 2007).

According to Lionnet (1985), sugarcane quality differs depending on the variety, region, climatic conditions, degree of maturity and the amount of extraneous matter (tops and leaves). Sugarcane contains approximately 13% sucrose, 16 % fibre and 69% moisture whereas the remaining 2% is said to be a mixture of gums, waxes, monosaccharides, inorganic material and inorganic acids (Lionnet, 1985). Gómez *et al.* (2014) mentions that tops and green leaves constitute approximately 8% of the sugarcane plant, whereas sheath and dry leaves constitute 20% and the remaining sugarcane stalk constitutes 72%.

Although the compositions of clean stalks reported by the different authors are very similar, it is noticeable that for tops and brown leaf, compositions are largely different suggesting that the nature of brown leaf and tops can be very different for varieties of sugarcane.

Table 2-1 presents the different compositions of sugarcane, leaves and tops from various authors. Leal and Hassuani (2000) reported the moisture content (on a wet basis) in sugarcane tops to be 82.5 g/100 g sample, 66.7 g/100 g sample for green leaves and 11.3 g/100 g sample for dried leaves. The ash content varied from 3.2 - 4.3 g/100 g sample (on a dry basis) and is 1.5 -2 times the value in the whole stalk. This suggests that the inclusion of such components in a diffuser could increase the ash content of the extracted juice.

Table 2-1: Typical analysis of cleaned sugarcane, sugarcane leaves and tops. Adapted from original table (Rein, 2007)

Brix (g/100 g sample)				
	Scott *et al.* (1978)	Ivin & Doyle (1989)	Birkett (1965)	Gil (2003)
Clean sugarcane stalks	16.7	16.6	15.3	14.2
Leaves	6.7	5.5	9.7	5.1
Tops	7.8	-	-	4.2
Pol (g/100 g sample)				
	Scott *et al.* (1978)	Ivin & Doyle (1989)	Birkett (1965)	Gil (2003)
Clean sugarcane stalks	14.8	15.1	12.9	11.9
Leaves	1.5	-	-	0.1
Tops	1.4	2.0	5.5	1.2
Apparent purity (%)				
	Scott *et al.* (1978)	Ivin & Doyle (1989)	Birkett (1965)	Gil (2003)
Clean sugarcane stalks	89	91	84	83.6
Leaves	19	-	-	2.7
Tops	21	36	57	24.1
Fibre (g/100 g sample)				
	Scott *et al.* (1978)	Ivin & Doyle (1989)	Birkett (1965)	Gil (2003)
Clean sugarcane stalks	12.8	12.5	13	12.7
Leaves	58.6	-	61	32.2
Tops	16.6	14.8	11.2	17.7
Moisture (g/100 g sample)				
	Scott *et al.* (1978)	Ivin & Doyle (1989)	Birkett (1965)	Gil (2003)
Clean sugarcane stalks	70.5	70.9	71.7	73.1
Leaves	33.6	-	-	63.5
Tops	77.7	79.7	79.1	77.2

Rein (2007) reported that the composition of sugarcane delivered to the factory (Table 2-2) is dependent on the sugarcane variety, presence of tops and brown leaf and other extraneous matter as well as the time of season, maturity of sugarcane and the delay between cutting and burning. The presence of tops and brown leaf in sugarcane is believed to reduce the purity of juice significantly. The leaves are said to increase the fibre content by 47% and the ash content by 62%. The tops are noted to have a smaller effect with a marginal increase in fibre and a 32% increase in ash content. Adding tops to clean stalk was calculated (using data from Scott *et al.* (1978) in Table 2-2 and was found to reduce juice purity by 0.3 units for every 1% tops. The presence of brown leaf and tops is said to reduce the juice purity by 5%. The extraction was achieved using the press method which is described by Lionnet (1996). Kent (2007) reported a 0.3 unit decrease in mixed juice purity for a 1% increase in trash by milling.

Table 2-2: Typical analysis of sugarcane delivered to the factory compared to clean stalk (hand cleaned and topped). Reproduced from Rein (2007)

Sugarcane sample	**Pol in g/100 g sugarcane**	**Brix in g/100 g sugarcane**	**Purity (%)**	**Moisture in g/100 g sugarcane**	**Fibre in g/100 g sugarcane**	**Ash in g/100 g sugarcane**
Green topped	11.5	13.6	84.6	65.2	21.2	2.5
Clean stalk-Green	14.7	16.2	90.8	71.6	12.2	0.4
Burned topped	13.6	15.4	88.4	70.2	14.4	1.1
Clean stalk –Burnt	14.0	15.6	89.6	71.3	13.1	0.8
Green untopped	10.6	13.2	80.4	65.5	21.6	2.8
Clean stalk- Green	15.0	16.5	90.9	71.3	12.2	0.4
Burnt untopped	13.4	15.5	86.4	69.8	14.7	2.1
Clean stalk -Burnt	14.5	16.2	89.4	71.3	12.3	0.4

According to Rein (1972) the most abundant non-sucrose components present in sugarcane are the monosaccharides, namely fructose and glucose which are termed reducing sugars. These components are said to be most prominent near the top of the sugarcane stalk. The contents of the reducing sugars are generally higher in sugarcane harvested during the high growth period. Polysaccharides, namely starch, cellulose, gums and dextran, are high molecular mass carbohydrates. They vary in concentration from 1 500 mg - 3 000 mg per kg dissolved solids depending on sugarcane variety (Legendre *et al.*, 1999). The concentration

of polysaccharides is said to be higher in tops and leaves than in stalk (Rein, 2007). The colour associated with tops and leaves is much higher than that of the sugarcane stalk. An increase in 1% tops or leaves is said to cause a 4% and 15% increase in colour respectively (observed in juice extracts from the cold digestion method).

Some sugar colour originates from the sugarcane itself, which contains natural plant pigments (Tarique, 2018). Such components include organic acids and waxes. Sugarcane variety is said to be classified according to the intensity of such colorants. Colour is said to vary along different parts of the sugarcane stalk substantially. These compounds are believed to decompose later in the sugar process, which results in enzymatic browning.

Lionnet (1985) highlighted that the compositions of tops and brown leaf are rather different to that of a green sugarcane stalk. In tops and brown leaf, the monosaccharides are believed to account for 50% of the soluble matter, whereas in sugarcane stalk it is approximately 15%. The sucrose content of these materials can be less than 1%. Furthermore, the sucrose content of brown leaf is approximately 1%.

Lionnet (1985) found that the addition of 15 % of brown leaf to clean green sugarcane stalk resulted in a juice colour increase of about 50 %. According to Lionnet (1985) colour bodies found in affinated sugar are said to originate from the sugarcane itself or may be formed during the process from coloured compounds originally present in the sugarcane or from colourless pre-cursors. Prabhakar *et al.* (2010) showed that a 550-1400 IU colour increase occurs for a 1 % increase in trash by milling. Approximately two thirds of the colour in raw sugar is believed to originate from phenolics and flavonoid groups, whilst the remainder is due to Maillard reactions (Lionnet, 1985). A Maillard reaction is defined as a reaction that occurs between amino compounds and reducing sugars, which produces melanoidins. The source of colour is plant derived but the melanoidins cannot form until the reactants are heated.

Rein (1972) deduced that the ash content of sugarcane has the greatest effect on the ash found in juices. The ash content in sugarcane is affected by the sugarcane variety. Tops and leaves are said to have a higher ash content in comparison to sugarcane stalks (Rein, 2007). According to Gómez *et al.* (2014), sugarcane trash contains approximately 2% ash. The

most significant inorganic material is known as silica. Potassium is the most abundant cation found in sugarcane followed by calcium, magnesium and sodium. The most abundant anion present in sugarcane is chloride followed by sulphate and phosphate (Rein, 2007).

Studies show that the burning of sugarcane also contributes to reduced purities and increased colour and ash contents. Reid and Lionnet (1989) showed that a 1.3% purity drop, and 41 ash % sugarcane increase is observed for burnt and topped sugarcane as opposed to manually cleaned sugarcane. In addition, juice extracted using milling methods with the presence of brown leaf contributed to 3% drop in purity, a 17% increase for ash on brix and 71% increase for colour content in mixed juice. Rein (2007) shows that a cleaned topped green stalk has a higher purity and lower ash content as compared to a burnt topped clean stalk (Table 2-2).

2.1.2 Sugarcane varieties

One of the main functions of the South African Sugarcane Research Institute (SASRI) is to develop improved sugarcane varieties for the South African industry (Parfitt, 2005). This is ensured through breeding, selection and release of sugarcane varieties that are adapted to the major agro climatic environments of the sugarcane producing area. Physiological characteristics of the varieties are said to be the determining factors for a variety's suitability.

Each sugarcane variety is represented and identified by a unique number e.g. N25 or N39 according to its characteristics. A detailed profile/information sheet can be obtained from SASRI for each sugarcane variety and includes details such as limiting features, soil suitability, agronomic characteristics, yield and quality. Such information allows sugar factories to identify what processing challenges may be faced with each sugarcane variety as well as what outputs to expect based on previous experiences over the years. The effect of sugarcane variety is said to be based on maturity, season, sugarcane yield, juice quality, suitability to growing conditions e.g. soil type, irrigation, regime season, ratooning potential, resistance to pests and diseases and adverse growing conditions (SASRI, 2019).

2.2 Types of solid-liquid extraction methods and methods used in the sugar industry

Different solid-liquid extraction methods are believed to result in different compositions of the various constituents extracted in sugarcane juice. The selection of the extraction method

is said to be highly dependent on the solid and solvent properties and quantities. The extraction method influences the cost of the system and will also affect the quality of the extracted juice that will impact on downstream processes. Conventional extraction methods employed in different industries were studied to obtain a general understanding. A key difference between these methods lies in the solid-liquid contacting patterns (Ming, 2007). According to Seader *et al.* (1998), solid-liquid extraction can be conducted under batch, semi-continuous and continuous conditions. Industrial equipment for solid-liquid extraction is designed for both batch-wise and continuous processing.

2.2.1 Single -stage batch extraction and batch extractors (Ahmed and Rahman, 2012)

In this process, the solid is contacted with a solute-free solvent until equilibrium is reached. The solvent may be pumped through a bed of solid and recirculated (Percolation) or the solids may be soaked in the solvent with or without agitation. Agitated vessels are usually used for batch extraction involving small particles that can easily be suspended in the liquid. Numerous types of agitators such as propellers, impellers and paddles can be used. The extraction time is dependent on the particle size, diffusivity of the solute in the solid matrix and the mass transfer coefficient. The latter depends on the flow pattern and mechanical energy input to the agitator. After equilibrium is reached, the solid is allowed to settle and the liquid phase is filtered or decanted. Percolators are used when solid consists of larger particles and is dense and difficult to maintain in a suspended state. The solvent is fed at the top of the vessel (usually under pressure to increase flowrate) and then percolates down the bed of solids, which is held within the vessel.

2.2.2 Counter-current extraction and counter-current extractors (Ahmed and Rahman, 2012)

This process involves solute-free solvent entering a system at the opposite end from the point of entry of the fresh solids. The solvent contacts the solids in the last extraction stage resulting in the lowest concentration of solute in the solvent phase at equilibrium in the last extraction stage.

The extract (solute rich solvent) leaves the system at the first extraction stage after contacting the solids that have just entered the system. Stage to stage flow of solvent moves in a direction counter-current to that of solids. The same solvent is used from stage to stage. Due to this, the solute concentration in the solvent increases while the solid moves from one stage to the next while the solute concentration in the solids decreases as the solids move in the opposite direction (Figure 2-2).

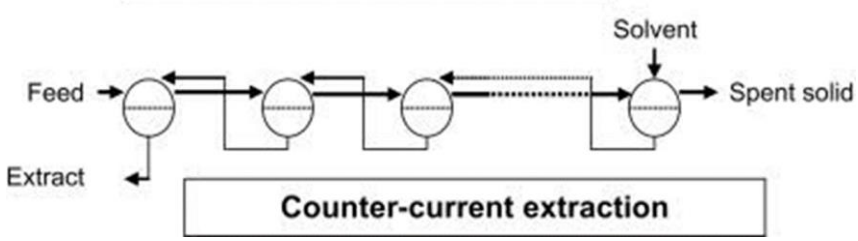

Figure 2-2: Basic principle of counter current extraction. Adapted from Food process engineering and technology (Berk, 2013)

Batch operating extractors can be operated as a counter-current cascade in a semi- continuous manner. Several vessels are connected in series. The solvent flows sequentially from one end to the next. Once the solid in the initial vessel is depleted, it is emptied, filled with fresh solid and moved to the end of the cascade. The second tank then receives the fresh solvent. Rearrangement is achieved by sometimes re-routing the fluid flow with a system of valves.

Belt and screw conveyors are converted to extraction equipment by adding a liquid circulation system (pump or gravity). For belt extractors, a horizontal belt conveys solid from one end to the next whilst solvent is introduced as a spray at the opposite end and collected under the belt. The solvent is pumped to the next spray nozzle and proceeds in this

manner to create counter-current contact. In screw extractors, the screw conveys the solid up the slope while the solvent percolates down in the slope. An industrial application that works on the principle of counter-current extraction is the well-known diffuser unit employed in sugar factories.

2.2.3 Sugarcane juice extraction methods used in the sugar industry

2.2.3.1 Cold digestion

The cold digestion or DAC (Direct analysis of cane) method (SASTA, 2009) is a standard method employed for decades by all sugar factories in South Africa and is used to analyse sugarcane. Several authors such as Davis and Barker (2013) and Barker and Muir (2010) carried out work relating to the cold digestion method in recent years.

Buchanan (1967) described cold digestion as a method which opens all cells within the sugarcane structure to release all dissolvable material and it is therefore believed that the method will extract more brix, especially non-sucrose as opposed to diffusion. An understanding of the cold digestion process can be established from early literature where authors such as Buchanan and Brokensha (1974) investigated the application of direct cane testing to the South African sugar industry.

The DAC (Direct cane analysis) method (SASTA, 2009) is carried out by taking a 1000 g shredded sugarcane sample and blending it with 2 litres of water in a high-speed digester for 20 minutes. The extract is transferred through a special assembling, which filters and cools the juice. The brix of the juice is measured at 20°C using a refractometer. The calculation of pol % sugarcane requires the quantity of juice in sugarcane as well as pol % juice to be known. An indirect fibre determination is carried out by drying 300 g of the same sugarcane sample at 105^0C for an hour. The moisture content is obtained by mass difference before and after drying. The fibre is calculated by deducting moisture and brix from the sugarcane. The natural fibre is taken as 1.25 times the amount of dry fibre and the former is deducted from sugarcane to obtain the juice content. Using this, pol of extract and pol in sugarcane is calculated.

Experiments were also performed to compare cold digestion versus hot digestion and cold digestion under different conditions (Buchanan and Brokensha, 1974). The digester was not water-cooled and temperatures around the 65°C mark was generally attained. Although the temperature could range between 50°C - 80°C depending on ambient temperature, the quantity and possibly the quality of fibre. A small degree of evaporation occurred during extraction and decantation. The experiments showed that at 74°C, an evaporation of 0.8% on sugarcane occurred. Tests were performed to illustrate the effect of time on pol % and brix % sugarcane. The maximum pol extraction of 99.4% was achieved at 20 minutes.

Conclusions drawn indicated that cold digestion is 1.5% short of the result obtained by hot digestion (boiling for 90 minutes). The shortfall may be attributed to incomplete extraction and partially to sucrose inversion. Brix extraction showed a substantial increase beyond 30 minutes. The tests conducted with water-cooled cold digestion (extract temperature maintained below 40°C) did not show such a large increase in brix. Reasons suggested for the large increase in brix observed in the standard procedures were attributed to the fraction of the sugarcane, which is normally insoluble going into solution at the elevated temperatures.

Tests comparing the standard cold digestion procedure with the water-cooled procedure showed that a 20-minute standard procedure brix did not experience any distortion. The results indicated that if the present design of cold digesters is to attain the pol extraction compared to hot digestion, longer extraction times and water-cooling will be essential. Water cooling would be required to avoid excessive evaporation losses and brix distortion linked to elevated temperatures due to longer extraction times. Buchanan and Brokensha (1974) concluded from their studies that extraction processes which occur during the DAC analytical procedures may, in fact, be duplicated on a much larger scale in a diffuser.

Davis and Barker (2013) indicated that the cold digestion method was bound to cause pol under-determination due to pol inversion during digestion at elevated temperatures. In addition, a further concern with the press method was the dilution factor. The method involves dilution of the residual juice in the bagasse by a factor of approximately eight. The brix of the bagasse extract was consequently very low at approximately 0.25 %. The value

was said to be close to instrument tolerances, especially those of brix refractometers which have a tolerance of ±0.05 % and therefore this increased the analytical uncertainty.

2.2.3.2 Press method

Lionnet (1996) investigated the effects of selected sugarcane and final bagasse characteristics on extraction using a sugarcane press and gave a basic understanding of how the press method functions. The experiments performed were said to simulate the milling process.

Fresh sugarcane stalks of a selected variety were topped and trashed manually. The stalk diameter at the top, middle and bottom, stalk length, stalk mass, number of nodes and internodes were measured and recorded. The stalks were then shredded in a Jeffco-cutter grinder. The experiments were performed using a pressure of 100, 150 and 200 x 10^5 Pa. Tests were performed using no addition of water and 300 g of water. The number of pressings tested were either 1, 2 or 3 times. The masses of cake and total juice extracted was measured. The extracted juice was analysed for brix and moisture.

The conclusions drawn included that the physical characteristics of the sugarcane stalks (stalk length, diameter and mass) have notable effects on moisture, brix, fibre and purity and thereby could have a significant influence on the extraction process. Differences were identified from different varieties, which was attributed to the fibre content and hardness of the stalks. The brix extraction was observed to increase as pressure increased for some varieties while some with certain varieties, an optimum was displayed. Brix extraction was also shown to increase as the pressure increased for the number of pressings for most varieties.

Barker and Davis (2013) carried out more recent studies on the press method where the press method was compared against other methods for bagasse analysis. It was of concern that the press method did not expose all unopened cells and merely squeezed the juice out which may have left the pol/brix-containing cells intact leading to underestimation of pol, brix and sucrose % bagasse. However, due to the long retention times, high temperatures and high extractions in diffusers, the percentage of unopened cells in the bagasse was likely to be negligible. Results indicated that the press method gave lower pol and brix % bagasse values

than the cold digestion method and that it underestimated the concentrations of the constituents of the bagasse.

2.2.3.3 Milling

One of the common juice extraction methods applied in industry was the process of milling, and many sugar factories to this date still make use of milling tandems. The simplest form of milling occurs in a 3-roll mill. The equipment is described as 3 grooved rollers turning at speeds of 3-7 rpm. The prepared sugarcane is squeezed between the rollers, which results in juice being forced out of the fibre. Therefore, the basic aim of the mill is to separate the juice from the fibre. However, the fibre has a natural tendency of always retaining its own mass of juice regardless of the pressure applied to it. Thin juice or water is poured onto the sugarcane fibre before crushing to dilute or displace the juice that is trapped. The term given to the water or juice is imbibition (SMRI, 2012).

Due to a single milling unit achieving undesirably low extraction rates, a typical milling unit in industry consists of 5 mills set up in tandem (Figure 2-3). The sugarcane is passed from mill 0 to mill 4 in series whilst imbibition is added to the fibre before mill 4. The thin juice from mill 4 is used as imbibition before mill 3 and so forth until mill 1 juice is combined with mill 0 juice, where no imbibition is used and the mixture which is mixed juice is sent for processing. In a mill, the fibre moves down the line from mill 0 to mill 4 and the juice moves in the opposite direction and is finally removed from tandem at mill 0 to mill 1. The final bagasse leaving the mill constitutes 50% moisture, 48% fibre and 2% brix. The final bagasse is used as a source of boiler fuel. The Mixed Juice is screened and thereafter contains 1% insoluble solids (SMRI, 2012).

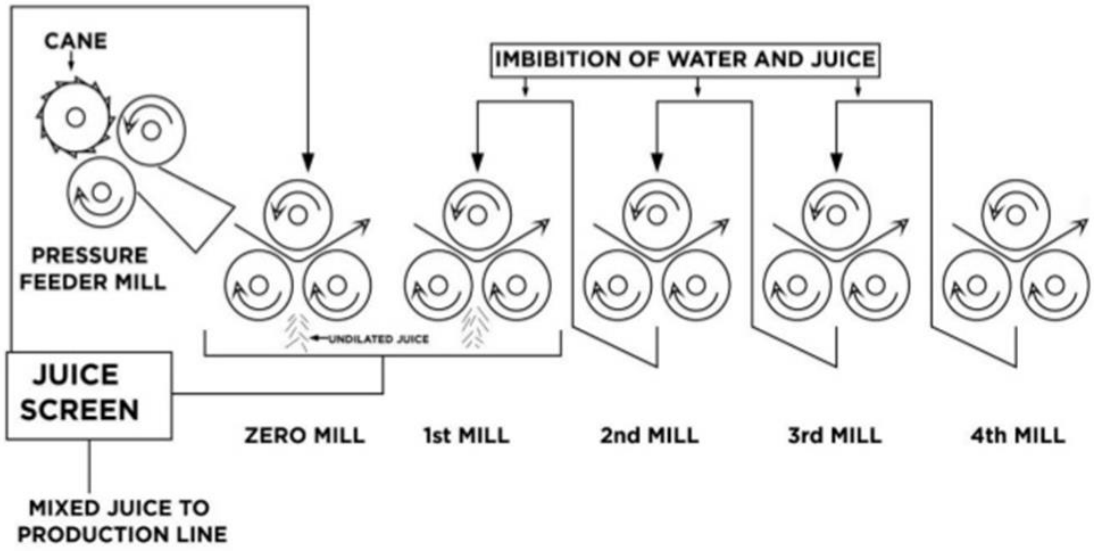

Figure 2-3: Sugarcane milling tandem schematic. Adapted from Report on process overview of Gobind sugar mills LTD (Venugopal, n.d:17)

Research and studies (Hemaida *et al.*, 1977; Reid and Lionnet, 1989; Kent, 2007; Muir *et al.*, 2009) have already been conducted on milling tandems with respect to green sugarcane processing since milling tandems were employed for juice extraction in the early days of sugar processing.

2.2.3.4 Diffusers - an application of counter-current extraction in a typical sugar factory

Although some investigations have been performed on diffusers in understanding the effect of brown leaf on sugar recovery and quality by Bernhardt and Pillay (2000) and Muir *et al.* (2009), the results were largely inconclusive for the purpose of quantifying the operational and cost impacts due to the challenges faced with carrying out trials on a full scale diffuser (changing sugarcane varieties) or simulating the process in a full scale diffuser. Since majority of the sugar factories have employed diffusers as their current method of sugarcane juice extraction, the proposed study therefore focused on reviewing work relating to diffusers in more detail, to determine the effect of processing green sugarcane with brown leaf in a diffuser, which is otherwise lacking. The lack of experimental work to date is due to the logistics around carrying out such trials in a commercial diffuser (Barker, 2017a). The limitations are attributed to difficulties in filling up the large diffuser with the same type of sugarcane, the lack of control over the variation of sugarcane entering the diffuser and the

difficulty in relating the brown leaf in sugarcane entering the diffuser to the mixed juice output (due to fibre retention times).

A typical diffuser (Figure 2-4) can be approximately sixty meters long by six meters wide (Rein, 2007). The vessel floor is made of a perforated plate over which a chain rides. The sugarcane is dragged along by the conveyor at a speed of 1 m/min. Below the perforated floor, the diffuser is usually divided into twelve stages. Each stage is comparable to a single milling unit. Press water and imbibition are added ahead of stage twelve, whilst thin juice from stage twelve is recycled to just ahead of stage eleven and continuing in the same manner until the juice is withdrawn from stage one. The flow of fibre is counter-current to the flow of juice. Juice is heated up to about 90°C at the feed end of the diffuser (Rein, 2007). Scalding juice is a term that refers to the portion of stage one juice that is tapped off and poured onto the incoming sugarcane to saturate and heat it. The scalding juice is recirculated through the heaters at the feed end of the diffuser at a rate of about 300% on sugarcane. The remainder of the juice can be referred to as diffuser juice, draft juice or mixed juice and is sent for processing.

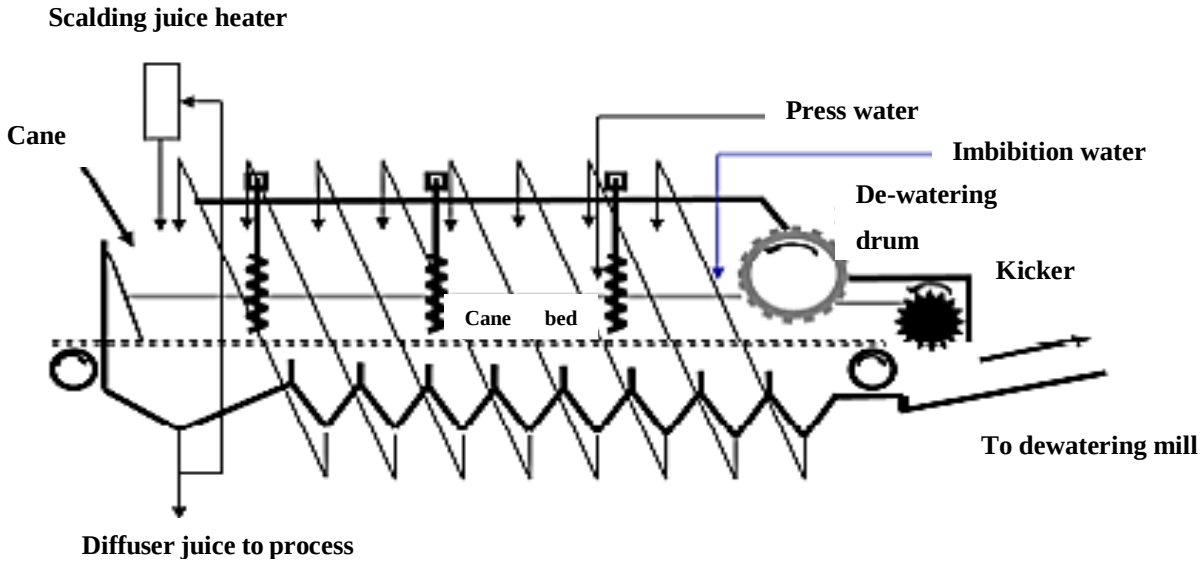

Figure 2-4: A schematic of a typical counter-current diffuser (SMRI, 2012)

The diffuser also has a discharge end sealed by a weighted drum which dewaters sugarcane to a certain extent. A spiked rotor called a kicker breaks off chunks of the hot fibre, which then falls onto a carrier that feeds the dewatering mill. Heat is applied by injecting steam

into the diffuser juice compartments to maintain a temperature of approximately 80 - 85°C to primarily improve the extraction and in addition to hinder bacterial growth (Rein, 2007).

The percolation through a fibre bed is an important factor in a diffuser. Lifting screws disturb the bed thereby preventing any packing that is expected to occur during the typical one-hour travel through the diffuser. The helical screws, which are installed vertically in the sugarcane bed, rotate and lift the sugarcane at several locations in the diffuser, including the point of press water return since the press water is said to contain sufficient fines to form an impermeable layer.

2.2.3.5 Typical BMA (Braunschweigische Maschinenbauanstalt AG) diffuser model

According to Hugot (1986), a typical BMA diffuser consists of a horizontal trough fitted with a bottom screen. A system of chains with a variable-speed drive serves to convey the feed through the diffuser. The model includes two rows of lifting screws in the front and middle extraction zones which are designed to improve juice percolation. In addition, a low-pressure de-watering unit is arranged in the de-watering zone. Feed enters the diffuser at ambient temperature and is heated to the desired extraction temperature by juice which is previously heated by heat exchangers with vapour or exhaust steam. The extraction temperature is maintained at a constant level by blowing steam into the space below the bottom screen.

2.3 Mechanism of extraction for sugarcane

The juice extraction process from sugarcane is believed to occur by two mass transfer processes in parallel. The first stage (a washing process) includes rapid extraction which involves the extraction of soluble matter in broken cells from the surfaces of the particles. The rate of transfer in this process is governed by the velocity of the liquid flow past the particles. The second mass transfer process is much slower and represents the mass transfer of the sucrose from the unbroken cells by diffusion.

Rein (2007) mentioned that dissolved molecules in solution will diffuse as a result of a concentration gradient. The diffusion will continue until an equilibrium is reached. If the raw material (e.g. sugarcane) is immersed in water, sucrose in the cells will diffuse across

the cell walls into the extracting liquid. This occurs due to the cell walls being denatured by heat. The process requires cutting the raw material into smaller pieces (shredding). The raw material is then mixed with water or juice that has a sucrose concentration that is lower than the cells of the raw material. The ratio of rate coefficients of the two mass transfer processes in a fully mixed environment is of the order 100, whilst in a packed bed where solid-liquid contact is lower, the ratio is closer to 50 (Rein, 2007). In a packed bed the higher juice velocity is believed to promote the rate of transfer and improve contact with particles thus reducing the amount of juice which has to be extracted by the slower diffusion process. In a commercial application, a counter-current diffuser is used. In this process, as extraction proceeds along the length of the diffuser, the concentration gradient is reduced and the concentration of non-sucrose in the extract increases. Rein (2007) recommends that adequate preparation of the sugarcane is required for the juice-bearing cells to be ruptured allowing for the juice in the sugarcane to be exposed to the extracting liquid.

Buchanan and Julliene (1969) suggested that most of the mass transfer during extraction occurs by washing which was evident by the rapid initial extraction of sucrose observed in the experiments conducted. The nature of extraction can therefore be described as a mass transfer process controlled by washing and diffusion in parallel. The extraction of larger particles is understood to occur by the control of molecular diffusion. Molecular diffusion is said to be more pronounced in a diffuser than in a milling tandem due to concentration gradient and the long residence times. However, mass transfer of soluble matter by molecular diffusion contributes to only a minor portion of the entire extraction cycle. The rate of transfer is believed to be positively influenced by higher temperatures, which allows for the intact cell walls to become permeable. Hence, the extraction rate and extraction are known to increase as the temperature increases. For efficient extraction to be achieved, the diffusion coefficient, particle size and diffusion time are still the determining factors even if the preparation of sugarcane is extremely fine.

2.4 Factors affecting juice extraction in diffusers

In addition to understanding the mechanism of extraction in a sugarcane diffuser, studies have been conducted in attempts to identify and quantify the various factors that affect the

diffuser-based extraction process. Studies have identified the most important factors that affect the extraction process as follows:

2.4.1 Effect of cane preparation

Good sugarcane preparation assists in achieving higher extraction and is considered the most important variable affecting extraction (Rein, 2007). For a higher extraction, sugarcane preparation in a heavy-duty shredder is recommended to allow for rupturing of the majority of the sugar-containing cells. The manner in which the sugarcane is prepared is also significant since all cells must have ruptured but with evidence of long fibres. The long fibres are believed to provide a stable and more open sugarcane bed to allow for higher percolation rates (Rein, 2007).

Payne (1968) highlighted that increasing the preparation index from 92 to 94 increases sucrose extraction by about 0.4%. Increasing the preparation index from 88 to 92 increases sucrose extraction by about 1%. Practically, the degree of preparation cannot be varied widely without altering the juice flow system within a diffuser. Several studies are in agreement that the intensive preparation of sugarcane increases extraction efficiency due to increasing exposure of the sucrose-containing cells to the extracting liquid. However, it is recommended that the preparation should not be too fine as this can lead to a phenomenon of flooding in diffusers, which is discussed later. Suggestions are made in the literature to prepare sugarcane using a Jeffco cutter-grinder although Lionnet *et al.* (2005) mention that it is not representative of the industrial preparation of sugarcane. A Jeffco prepares fine sugarcane, whilst a Waddell shredder is said to prepare coarse sugarcane, which is more representative of the sugarcane preparation in industry.

Lionnet (1985) showed the differences between the mean values of the coarse and fine preparations were statistically significant in the case of brix and total phenols. In the case of brix, which in these experiments was between 85 and 90% of the sucrose, the fine preparation resulted in more than 90% of the material being available through the washing mechanism, during the first 4 to 5 minutes of the 60-minute extraction. Coarse preparation reduced this amount to 85% showing that, even then, the major proportion of the sucrose is extracted in the washing step. Rein (1972) also showed that higher extraction is achievable with finer bagasse preparation.

2.4.2 Effect of sugarcane residence time

According to Rein (2007) the longer the sugarcane spends in the diffuser, the longer it is contacted with the liquid and therefore the better the extraction. Sugarcane residence or retention time is therefore a fundamental process variable to consider. Rein (1972) calculated that a change in residence time of just 5 minutes would change the overall brix extraction by 0.5 in a diffuser with a normal bagasse retention time of 44.1 minutes. The effect of sugarcane residence time on extraction is presented in Table 2-3 which shows that an increase in residence time beyond 50 minutes only increased the extraction by 1%. Typically, commercial diffusers sugarcane retention times vary for different types of diffusers and can vary between 60 to 90 minutes (Matthesius, 1977; Hugot, 1986) .Very long retention times can result in inversion of sucrose and formation of colour and therefore the effect of increasing retention times to improve extraction must be carefully considered to avoid such consequences.

Table 2-3: The effect of sugarcane residence time on % extraction for diffusers. Adapted from original source (Rein, 2007)

Sugarcane residence time (minutes)	**Screen area ($(m^2/h)/t$ fibre)**	**Extraction (%)**
87	13	98
67	10	97
54	8	96

Retention times varied from 25 minutes to 50 minutes for studies using percolation and submerged columns (Buchanan and Julliene, 1969). Pamu (2012) whose studies involved simulating a four-stage counter-current unit used residence times of 15-45 minutes. Pol (1957) made use of a continuous screw type conveyor simulated diffuser pilot plant and used a residence time of 7 hours. Ming (2007) looked at a pilot-scale counter-current extractor and used residence times of 1-1.5 hours. Lionnet (1985) used a residence time of 60 minutes in his studies involving a batch mixed extraction process.

2.4.3 Effect of imbibition % fibre

The quantity of imbibition water added in relation to the quantity of sugarcane also affects the extraction since the extraction process in a diffuser involves concentration gradients as a driving force for mass transfer. The use of large quantities of water is expected to create much larger concentration gradients, which can be beneficial in the extraction process. Rein (2007) identifies the imbibition rate as a factor that affects diffuser-based extraction. As with any other solid-liquid extraction process, the more extracting liquid added, the easier the extraction. The amount of imbibition water added is related to the quantity of fibre being processed.

South African mills have been reported to use imbibition rates of over 400% on fibre with consequent extraction benefits while some diffusers have operated on low rates such as 200 % imbibition on fibre (Rein, 2007). A recommended range for imbibition % fibre is not given in literature since this depends on the diffuser setup. Imbibition % fibre rates used in industry as well as their effects on % extraction are presented in Table 2-4. The amount of imbibition that can be added is affected by the percolation rate (discussed later), the position of the sprays and the flow patterns in the diffuser.

Table 2-4: The effect of imbibition % fibre on % extraction for diffusers. Adapted from original source (Rein, 2007)

Imbibition % Fibre	**250**	**300**	**350**
Case 1: High extraction	97.6	98.0	98.3
Case 2: Low extraction	94.3	95.0	95.4

These values can only be applied to counter-current extraction processes. The setup of a counter-current system on a pilot scale is challenging due to the complexity of the design. In addition, when setting up any other system, one will have to consider the amount of extraction that can be achieved and the concentration of constituents in the extract, since a very high imbibition may be needed to achieve the same extent of extraction as observed in a counter current system. However, the resulting constituent concentrations may be prohibitively low.

Lionnet (1985) used 375% water to sugarcane rate for a batch mixing pilot plant. Rein (1972) used 450% water to fibre rate in studies conducted using a batch mixing pilot plant. Ming (2007) used a water to sugarcane rate of 200% in his studies using a continuous screw type counter-current conveyor pilot plant.

2.4.4 Effect of percolation rate

The velocity of the liquid flowing past the sugarcane particles affects the mass transfer process. Suggestions are made to improve the degree of contact between the liquid and solid, which is determined largely by the percolation rate of the liquid through the bed of the sugarcane. The maximum percolation rate is therefore a significant variable that must be considered. Percolation rate is the rate of application of liquid to the top surface of the bed, without flooding it, expressed as m^3/min per m^2 of bed area. Studies show that percolation rates measured in laboratory diffuser columns were generally higher than those found in commercial diffusers (Rein, 1972).

For full-scale diffusers, the range is 0.1-0.2 m^3/m^2.min (Rein, 2007). Percolation rates in laboratory columns, however, range between 0.3 and 1.0 m^3 /m^2.min and are largely dependent on the fibre packing density (Lionnet *et al.*, 2005). This may be due to laboratory tests being performed in small diameter columns under flooded conditions thereby reducing channeling (Lionnet *et al.*, 2005). Percolation rates are said to be lower with a finer sugarcane preparation and a more compact bed and tend to decrease along the length of a diffuser.

Percolation rates determine where the inter-stage juice sprays should be located for juice to appear in the correct tray at the bottom of the diffuser (Rein, 1972). Investigations performed by Loubser and Barker (2011) showed that sugarcane varieties also have an effect on percolation rates since the sugarcane variety determines the packing behaviour and degree of bed compaction. Results from the experimental work are presented in Table 2-5. The actual properties of sugarcane depend on a multitude of parameters such as age, rainfall, sunlight and season. Significant differences between the varieties were found at the 95% confidence interval (Loubser and Barker, 2011). Bed heights in a diffuser range from 1.1- 1.8m. A higher bed height is said to cause unstable percolation conditions and therefore factories prefer operating at lower bed heights.

Table 2-5: Effect of sugarcane variety on percolation rate in order of perceived diffuser performance by factory personnel. Adapted from original source (Loubser and Barker, 2011)

Ranking by factory	**Sugarcane variety**	**Percolation rate (m/min)**
1	N14	1.01
2	MN1	0.81
3	N36	0.72
4	N25	0.48
5	N32	0.48
6	N38	0.52

Loubser and Barker (2011) performed work on a Perspex column to investigate percolation rates. A Waddell shredder was used to prepare the sugarcane and the post percolation bulk density of the sugarcane was 460 -510 kg/m^3. The mass of sugarcane used was 6 kg while the bed height was 0.4 to 0.5 m. The percolation rate was 0.5 to 1.1 m/min. Lionnet *et al*. (2005) studied percolation through prepared sugarcane beds on a lab-scale showed percolation rates varying from 0.06 to 0.26 m/min. Reference was made to work carried out by Love and Rein (1980). The two authors had also performed trials in a pilot column. Percolation rates ranged from 0.15 to 0.32 m/min. All abovementioned ranges correspond to values that are higher than commercial diffuser percolation rates.

Air trapped in the bed is said to reduce the percolation rate significantly (Buchanan and Jullienne, 1969). To improve percolation, recommendations are made to fill the bed upwards from the bottom to displace the air and prevent channeling. However, a limitation exists since the percolation rate cannot be measured due to the lack of gravitational flow in the column.

Flooding is the term referred to when the upper limit of the percolation rate is reached. This is when more liquid than can percolate through the sugarcane bed is introduced to the top of the bed surface. A number of operational problems arise from flooding, such as washing sugarcane out of the feed and discharge ends of a diffuser and destruction of the concentration gradient in the diffuser, significantly reducing extraction. Rein (1972) showed that flooding occurred at lower liquid throughputs with more finely prepared sugarcane. It was also indicated that flooding occurs at low flowrates with an increase in fibre density (inter-particle space is not large enough to permit flow), high temperature (due to softening of the fibres which results in an increase in packing density) and high beds of prepared sugarcane (Rein, 1972). When a diffuser is operated at optimum liquid flowrates, a suggestion is made to avoid flooding by decreasing the throughput and imbibition of a diffuser during operation (Rein and Ingham, 1992).

Rama *et al.* (2006) suggests that permeability in the sugarcane bed can be increased by removal of suspended solids which occur when high loads of suspended solids enter the factory due to high soil loads. Such occurrences are heightened during rainy weather conditions, especially upon delays between harvest and milling and depends on loading practices (Rama *et al.*, 2006). Several factors were identified to affect flooding including the season, brown leaf, tops and sugarcane variety. An uneven sugarcane bed will also cause flooding due to channeling and non-ideal flow. Several authors, including Boote (2010), Rein and Love (1980) as well as Lionnet (1985), are in agreement regarding the effects of percolation on extraction as discussed above.

Results obtained from column percolation tests (Figure 2-5) carried out at the Sugar Milling Research Institute (SMRI) in a glass column showed that the maximum percolation rate decreases exponentially with time as the sugarcane compacts (Jensen, 2013).

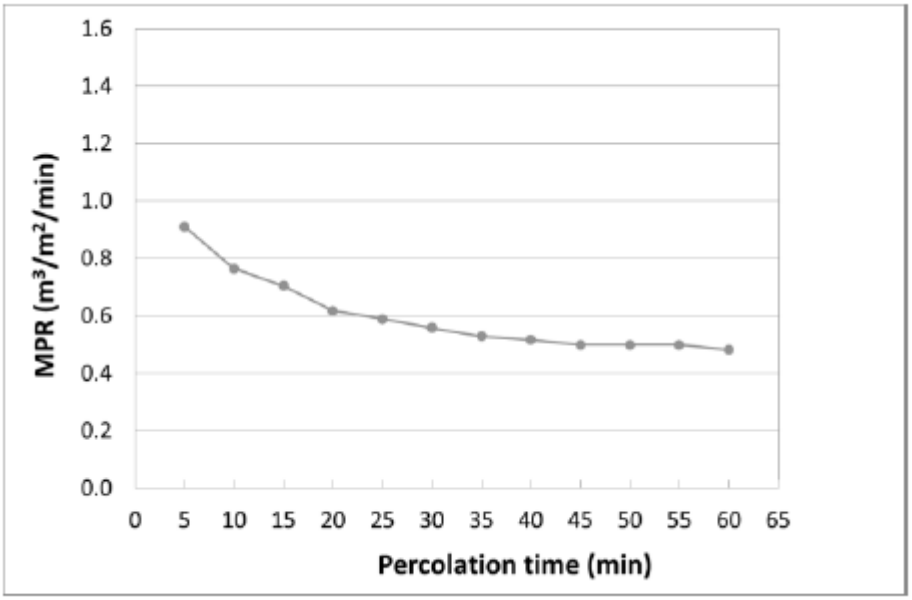

Figure 2-5: Maximum percolation rate (MPR) as a function of time performed in a column experiment (Jensen, 2013)

2.4.5 Effect of bed height

The bed height is also a factor that is said to affect extraction in a diffuser. Rein (1972) noted that lower instances of flooding were observed with lower percolation rates with higher packed bed heights. It is assumed that higher bed heights are associated with higher fibre densities. Therefore, the effect of the bed height can be explained with respect to density packing. Since flooding was observed to begin at the bottom of the bed (region of greatest local packing density), the result confirms the assumption. In addition, a higher temperature is believed to result in greater fibre densities since softening of the fibres takes place leading to a greater degree of compaction (Rein, 1972).

According to a colloquium (SMRI, 1978), it was suggested that a greater bed height results in the material at the base being more compacted which causes the fibre density to increase down the bed, thus inducing flooding. Furthermore, the quantity of air trapped in the bed was shown to have a negative effect on the percolation rate as a result of channeling of the extracting liquid which reduces contact with the solid. It was explained that in some of the trials conducted by various parties, filling the bed up from the bottom with juice before testing resulted in the percolation rate being higher. This was due to the upward flow of juice

displacing air in the bed. With a bed density that is uniform, a higher percolation rate is observed. It was also concluded that the influence of lifting screws was very localised since bagasse restores to its previous state after passing through screws.

2.4.6 Effect of bulk density

Bulk density is generally defined as the mass of solid per unit volume. This factor is important as it is said to dictate the volume required for a given sugarcane residence time and affects the percolation rate through the bed. The higher the density, the lower the percolation rate. Bulk density depends on particle size and sugarcane preparation; a finer sugarcane preparation is said to have a higher density. Bulk density of shredded sugarcane depends on sugarcane preparation and compaction and typically varies from 250 to 350 kg/m^3 (dry) on a conveyor at up to 0.5 m depth (Walsh, 1998). According to Hugot (1986), the average bulk density for wet shredded sugarcane in a diffuser is 500 to 600 kg/m^3. This means that the average apparent volume occupied by one ton of sugarcane varies between 1.67 to 2 m^3.

Bulk density used in various experiments ranges between 250 and 560 kg/m^3 (wet prepared sugarcane). Work carried out by Love and Rein (1980) in which sugarcane was prepared with a Jeffco cutter-grinder showed sugarcane densities ranging from 430 and 560 kg/m^3 (post percolation) while a bed height of 150 cm was used. A Waddell shredder was used to prepare the sugarcane where Loubser and Barker (2011) performed work on a Perspex column to investigate percolation rates. The post percolation bulk density of the sugarcane was between 460 -510 kg/$m^{3.}$ The mass of sugarcane used was 6 kg while the bed height was 0.4 to 0.5 m. The percolation rate was 0.5 to 1.1 m/min. Finer particle sizes are said to give a higher bulk density. The bulk density is said to affect the percolation rate by an inverse relationship. It is also important since it is the determining factor that governs the volume of sugarcane required for a given sugarcane residence time (Rein, 2007). Due to this effect, it has been established that the sugarcane fibre density is a factor to be considered in the pilot extractor experimental work.

Barker (2017a) investigated the effect of unburnt sugarcane with brown leaf on percolation rates and sugarcane density. It was found that the effect of unburnt sugarcane with brown leaf increased the percolation rate by 50% compared to burnt sugarcane and decreased the

density by 40% compared to burnt sugarcane. Prabhakar *et al.* (2010) showed that the bulk density of loose dried leaves ranged between 50-65 kg/m^3. In the pilot extractor design, consideration must be given to the bulk density of green sugarcane and brown leaf since it is likely that these components will have a lower bulk density and will occupy a larger volume. Some studies performed showed no clarity in whether the densities stated are reported on a wet or dry basis. The densities are expected to differ as a result of the manner in which the sugarcane was prepared. It is therefore recommended that for the pilot extractor experiments, the sugarcane density should be determined by experimental trials. Consideration must be given to the fact that inclusion of green sugarcane stalk with brown leaf will result in lower densities and thus greater shredded sugarcane bed volumes.

From the literature reviewed, recommended bulk density ranges of green sugarcane stalk with brown leaf cannot be confidently suggested. It is recommended that the bulk density of shredded green sugarcane with brown leaf be measured experimentally before designing the extractor. The sugarcane mass must also be considered in the design as this determines the capacity of the pilot extractor and its size. Consideration should be given to what can practically be achieved in terms of ease of handling of sugarcane during factory validations. A reasonable quantity (mass) of shredded sugarcane sample must be used that will enable a good representation of the sugarcane in a factory diffuser and that will also minimise the dilution effects on the juice from the extractor.

2.4.7 Effect of temperature

High temperatures in diffusers are considered advantageous as they increase the rate of extraction by higher molecular diffusivity (increased reaction kinetics) and by lowering the liquid viscosity. In addition, high temperatures denature the protein lining of the cell walls and increase the permeability of the unbroken cells (Rein, 1972). The diffuser temperature is typically kept above 75 °C to control microbial activity.

The dependence of extraction on temperature and degree of bagasse preparation is shown in Figure 2-6 for an experimental trial performed by Rein (1972) in a packed bed column which can be found in more detail in section 7 .Rein(1972) made use of P1 (first mill bagasse), P2 (first mill bagasse fed through a mill), P3 (first mill bagasse fed through a mill with a coarse screen over the outlet), P4 (first mill bagasse fed through a mill with a fine screen over the

outlet) in his experimentation. Rein (1972) varied the temperatures (60°C, 70°C and 85°C). An increase of 0.2% in extraction was observed when the temperature was increased by 5 °C from 75 to 80 °C (Rein, 1972). Graphs showing the effect of temperature and bagasse preparation on % brix extract are shown in Figure 2-6.

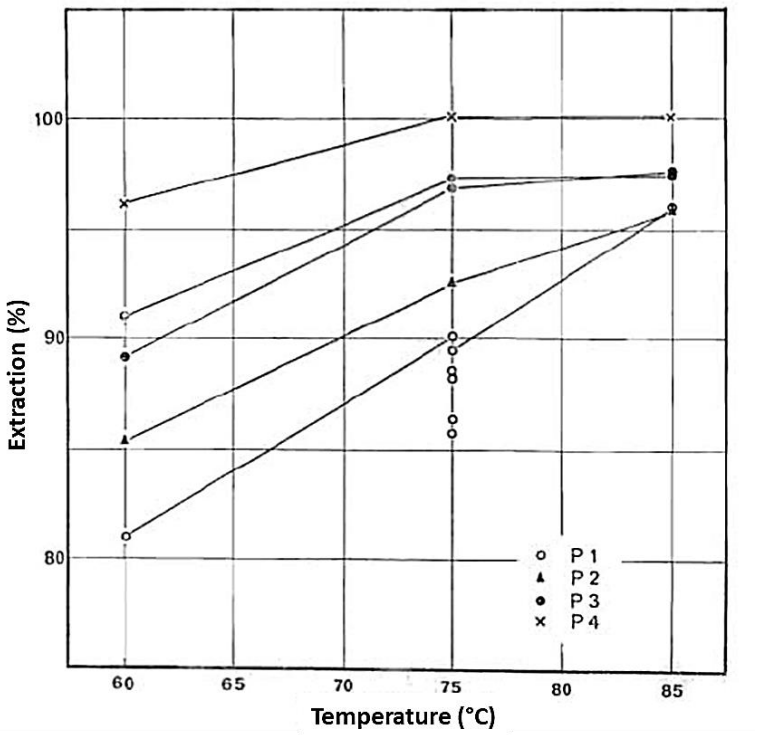

Figure 2-6: Dependence of extraction (%) on temperature and degree of bagasse preparation after 50 minutes. Adapted from original source (Rein, 1972)

Figure 2-7 and Figure 2-8 depict the pol and brix on % sugarcane for experiments performed by Lionnet (1985) using extraction achieved under equilibrium conditions in a batch laboratory process.

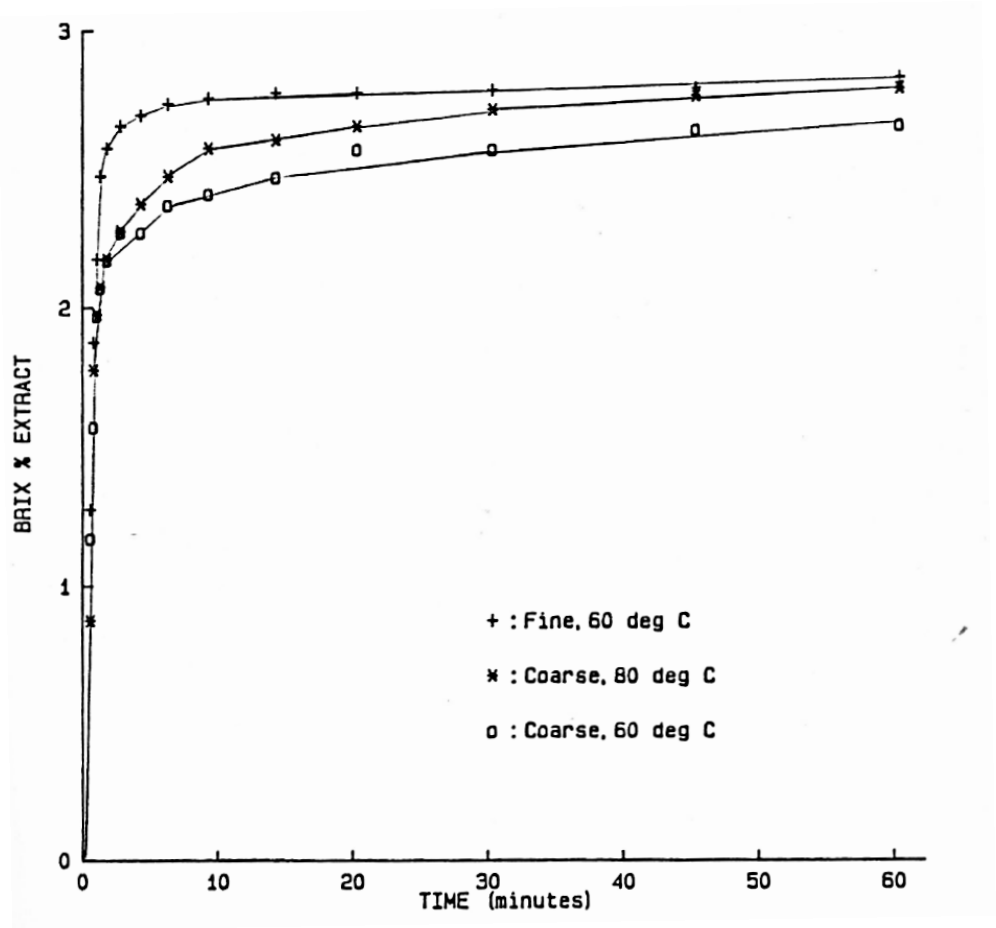

Figure 2-7: The brix % extract achieved with varying temperature and preparation (Lionnet 1985)

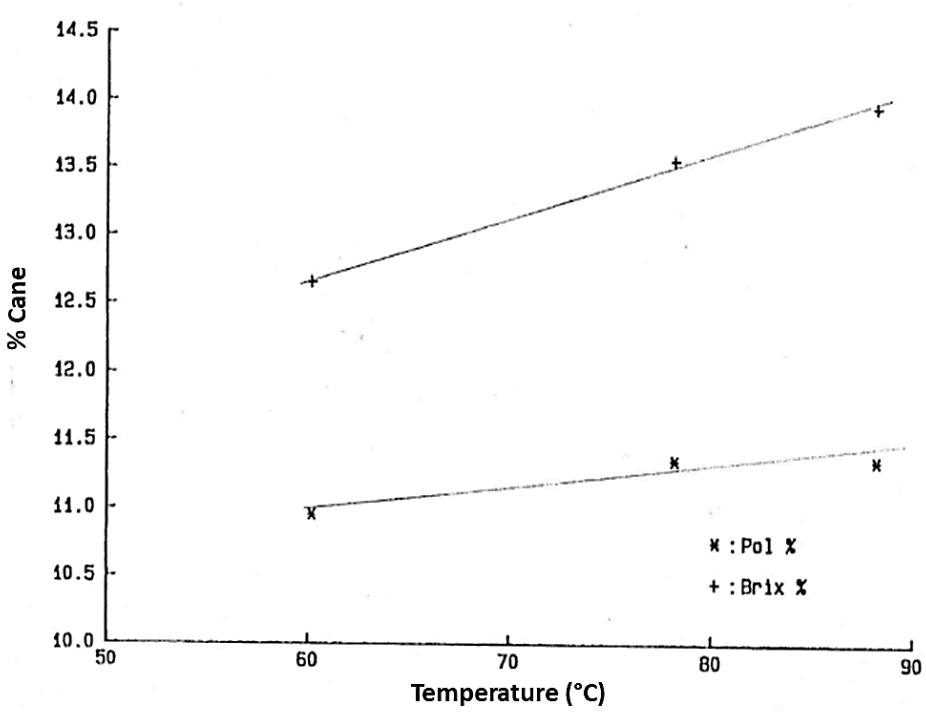

Figure 2-8: The increasing brix % and pol % on sugarcane achieved with increasing temperature (Lionnet, 1985)

Lionnet (1985) deduced that temperature had a marked effect on the absorbance of light measured by a spectrophotometer both in visible and ultra-violet regions (Figure 2-9). The experiments he conducted showed that the effect of temperature is about 5 times greater on absorbance than it is on brix. The temperature was observed to increase juice colour significantly without increases in pol extraction. Graham *et al*. (1968) concluded from their studies that the conditions of pH and temperature within a diffuser have a much greater effect on the amount of reducing sugars than the other constituents present in a diffuser juice since pH and temperature are two of the variables that affect the enzymatic breakdown of sucrose. High temperatures with low pH are said to promote inversion. An interesting observation is that Lionnet (1985) had chosen to add linear trend lines through only three data points. On inspection, there appears to be an increasing trend but with a lack of replication, no definite result can be concluded.

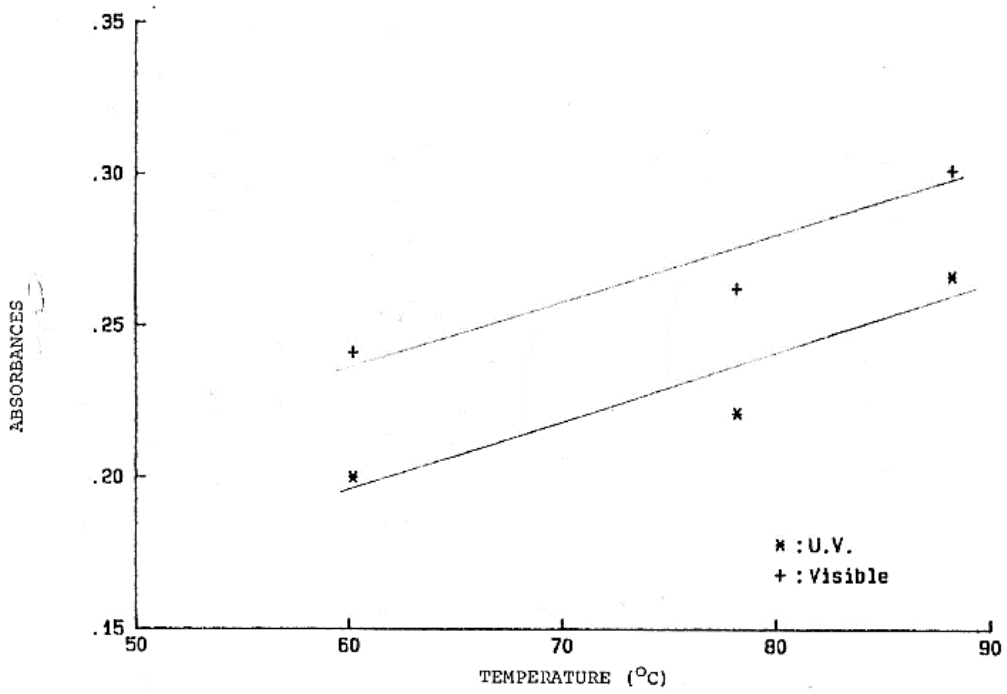

Figure 2-9: The absorbance of light measured in extract achieved with varying temperature (Lionnet, 1985)

Temperature is identified as a significant factor in the extraction process for two reasons; firstly, higher temperature promotes the rate of mass transfer by reducing viscosity and secondly, it denatures the protein lining of the cell walls, which increases the permeability of the unbroken cells. This is said to enhance molecular diffusion. The optimum temperature lies between 70 and 85°C. Temperatures below 70°C are said to favour microbial activity. Higher temperatures, which may favour extraction, can lead to inversion and higher colour formation in juices and for this reason, are generally avoided. The most common method used to maintain uniform and stable temperature control includes the design of a heating jacket around the extractor (Lionnet, 1985; Rein, 1972).

2.5 Nature/composition of mixed juice

2.5.1 Mixed /Draft juice composition

Sugarcane juice, extracted by the various methods, is understood as being a complex liquid medium containing numerous organic and inorganic constituents in soluble, suspended and colloidal forms (Saska *et al.*, 2010). The juice is commonly referred to as mixed or draft juice after passing through the diffuser extraction process. Walford (1996) studied the composition of mixed juice extracted from sugarcane; the results are presented in Table 2-6, Table 2-7 and Table 2-8.

Table 2-6: Typical concentration ranges of components in mixed juice. Adapted from source (Walford, 1996)

		g/100 g brix
Sugars	Sucrose	81-87
	Reducing sugars	3-6
	Oligosaccharides	0.06-0.6
	Polysaccharides including gums & dextran)	0.2-0.8
Salts	Inorganic salts	1.5-3.7
Organic non-sugars	Organic acids	0.7-1.3
	Amino acids	0.5-2.5
	Dextrans	0.1-0.6
	Starch	0.11-0.5
	Gums	0.02-0.05
	Waxes, fats, phospholipids	0.05-0.15
	Colourants	0.1
Insolubles	Sand, bagasse etc.	0.15-1

Table 2-7: Composition of carbohydrates in mixed juice. Adapted from source (Walford, 1996)

Carbohydrate	Units		Concentration
Monosaccharides	g/100 g sample	Glucose	0.26-0.33
		Fructose	0.26-0.33
Disaccharides	g/100 g sample	Sucrose	9.6-10.9
Oligosaccharides	g/100 g brix	1-Kestose	0.26-0.33
		6-Kestose	0.03-0.05
		Neo-Kestose	0.01-0.4
		Theanderose	0.01-0.4
Polysaccharides	g/100 g brix		0.3-1.3

Table 2-8: Composition of ions in mixed juice. Adapted from source (Walford, 1996)

Constituent		Concentration g/100 g brix
Cations	Potassium	0.77-1.31
	Sodium	0.01-0.04
	Calcium	0.24-0.48
	Magnesium	0.10-0.39
	Iron	0.006-0.04
	Aluminium	0.005-0.17
	Copper	0.002-0.003
	Zinc	0.003-0.012
	Manganese	0.007
	Cobalt	0.00007
	Silicon	0.016-0.101
Anions	Chloride	0.16-0.27
	Phosphate	0.14-0.40
	Sulphate	0.17-0.52

Sugarcane quality has a considerable effect on extraction in diffusers since impurities present in the sugarcane can also be extracted in the juice. The nature and composition of mixed juice has been reviewed in this study. This provides a basis for identifying the key constituents in the extractor juice to determine if the juice is representative of diffuser juice. Important constituents that characterise mixed juice obtained from a diffuser according to literature include sucrose, reducing sugars (fructose and glucose), ash, colourants, inorganic acids, cations and anions.

Ghada *et al.* (2009) mentions that sugarcane juice has a pH range of about 4.9 - 5.5. The juice has an opaque appearance attributed to the presence of components such as silica and colloidal substances like waxes, proteins, gums and starch. These components add turbidity to the juice. Chlorophyll and anthocyanin (from the sugarcane rind), saccharetin (from fibre) and tannins (from the bud and tops) are all colour components present in the sugarcane and are said to pass through with the juice on extraction.

Rein (2007) indicated that the two most marked effects of diffusion on juice quality are the higher colour and low suspended solids. The colour depends on the cleanliness of the sugarcane and the temperature in the diffuser. Further findings from the study conducted by Mullapudi (2010) were that the bagasse in the diffuser was shown to have filtered the juice. The suspended solids in the mixed juice obtained from the diffuser were approximately 80 ppm while in the milling tandem it was 2000 ppm. However, the diffuser juice was noted to be of a higher colour than from a milling tandem. Diffuser juice was concluded to have lower unknown losses (0.06% on sugarcane) due to the higher temperatures that minimise microbial activity.

Diffuser juice is said to have lower lactic acid levels as a result of reduced microbial activity. Furthermore, diffuser juice is said to have a slightly lower purity due to higher extraction. The suspended solids are reported to be 0.1 g/100g in juice from a fixed screen diffuser. Juice colour is directly proportional to colour in sugarcane varieties. The leaves and tops in sugarcane are major contributors to colour development in juices. A 10-20% colour increase is observed in diffuser juices obtained compared to mill juice (Rein, 2007).

Some impurities can lead to the loss of sugar in molasses. Apart from sucrose being extracted, a host of impurities can also be extracted in the sugarcane juice. Several impurities can contribute to considerable difficulty in back-end factory operations. Reducing sugars (RS) are the most abundant impurities present in the juice. Graham *et al.* (1968) showed that the ratio of reducing sugars to sucrose concentration in a full-scale diffuser decreases from the feed end to the discharge end. Rein (1972) suggests that a reason for this occurring is that the reducing sugar/pol ratio is higher from the last stage where imbibition water is added than in the juice from the penultimate stage. The water tends to elute the reducing sugar molecules from the bagasse. From this evaluation, it is suggested that reducing sugars appear to be extracted at a more rapid rate than sucrose. An explanation for this occurring in a once-through system is that initially both sucrose and reducing sugars are extracted at the same rate due to the washing mechanism being operative whilst later on reducing sugars are extracted more rapidly due to the diffusion process as a result of their smaller molecular size (Rein, 1972). In a re-circulated system, Rein (1972) describes a "chromatographic column effect", where smaller molecules are preferentially absorbed by the bagasse causing an initial drop in reducing sugar/brix ratios. The ratio of reducing sugars to ash plays an important role in sugar recovery. It is well known that the solubility of sucrose is decreased in the presence of reducing sugars while inorganic salts tend to increase the solubility.

Composition ranges relevant in this study, as presented in Table 2-6 for a typical mixed juice on % brix includes; sucrose (81-87%), reducing sugars (3-6%), inorganic salts (1.5-3.7%) and colourants (0.1%). It is expected that the extractor juice will be more dilute in comparison to a diffuser mixed juice. This should be taken into account when considering analytical measurement techniques as well as instrumentation chosen for installation on the extractor. Many of the constituents found in sugarcane juices are rather complex and difficult to quantify and, for this reason, it is suggested that the constituents be selected on the basis of being able to provide meaningful results with ease of measurement. For this reason, it is suggested that extracted juice used in the experimental runs be analysed for brix, sucrose, reducing sugars, ICUMSA colour and conductivity ash. The direct analysis of cane (DAC) method should be used as a reference to determine the maximum extraction obtainable from sugarcane since the method of cold digestion (Buchanan, 1967) is designed to open all cells within the cane structure to release all dissolvable material and it is therefore believed that the DAC method extracts more brix and non-sucrose than diffusion.

2.6 Suitable analytical methods for analysis of components present in extracted sugarcane juices

A number of appropriate and recommended analytical laboratory methods pertaining to the sugar industry have been reviewed to select the most suitable method for the sample analysis for the proposed work. These analytical methods reviewed only pertain to the components present in mixed juice that will be focused on in the study namely brix, sucrose, reducing sugars (fructose and glucose), conductivity ash and colour. The suitable methods include Gas chromatography, high liquid performance chromatography, Near-infrared spectroscopy and other sugar specific standardised analytical methods. Factors taken into consideration include availability of instrumentation, detectability ranges, precision, accuracy, time, cost and labour resources as well as the nature of the constituents in the sugarcane juice.

2.6.1 Fructose, glucose and sucrose analysis

HPLC (High-performance liquid chromatography) and GC (Gas chromatography) are both methods of separation of compounds from a mixture (Just chromatography, 2012). HPLC applies to constituents that are fluids while GC is used when the compounds are gaseous or vaporised during separation. Both methods work on the same principle whereby heavy molecules flow slower than lighter molecules. According to (Just chromatography, 2012), NIR (Near-infrared spectroscopy) has also become a popular new alternative to the two abovementioned methods. Walford *et al.* (2004) conducted an overview of chromatographic techniques used in the sugar industry for over 30 years such as HPLC and GC. These methods were described as precise methods which were used for sugarcane payment as well as factory control. The review for the proposed study was not to intensely describe the techniques in great detail but rather to provide a brief description of the method and review the suitability of the methods to the proposed work.

2.6.1.1 GC analysis

The method is used to mainly check the purity of a substance and can help identify a substance. Two phases exist, namely the mobile phase and stationary phase. The mobile phase is usually an inert gas while the stationary phase involves the use of a polymer or a layer of liquid on an inert solid base (Just chromatography, 2012). Costa and Conte-Junior (2015) mention that GC is more suitable for analysis of volatile organic compounds in

complex matrices whilst Walford *et al.* (2004) noted this as one of the drawbacks of the method. GC is said to provide good sample resolution and sensitivity but is not widely used for carbohydrate analysis due to the fact that the carbohydrates have to undergo prior derivatization to make them volatile (Costa and Conte-Junior, 2015). The review of studies (Walford *et al.*, 2004) indicated that the method was rendered suitable for determination of sucrose in mixed juice and proved to be much more precise and less time consuming as opposed to the older methods. Some of the advantages that came out of the review study (Walford *et al.*, 2004) was that the GC method involved tedious sample preparation, the use of toxic derivatisation chemicals and that the derivatisation of samples could give erroneous results. According to the SASTA lab manual (2009) this is an official method used in the sugar industry, applicable to all factory juices and is used to determine the glucose, fructose and sucrose contents of a sample.

2.6.1.2 HPLC analysis

The method is used to analyse and identify individual components in a mixture. Columns and high pressure are used which is said to allow for movement of the constituents in the mobile face and in addition allows for the movement of the analyte in a densely packed column (Just chromatography, 2012). According to American laboratory (2019), HPLC can make use of a PAD (pulsed amperometric detector) which is a pulsing voltage waveform that charges the electrodes, detects the analytes, clears the electrode and restores the surface. The waveform ensures that the surface is always clean, and the results are reproducible. PAD allows quantification down to the picamole levels thus reducing loss of sensitivity and specificity as seen with carbohydrates using UV and refractive index methods (American laboratory, 2019). Costa and Conte-Junior (2015) indicate that HPLC has gained importance in the analysis of carbohydrates and organic compounds due to the speed, sensitivity, selectivity and reliability. According to Ceutics (2014), the sample used in the HPLC method must be soluble in the solvents used for analysis. HPLC is said to impart a greater flexibility and range of application than GC (Ceutics, 2014). (Walford *et al.*, 2004) described the HPLC method as a logical successor to the GC method due to the lack of needing to derivatise a sample. However, when the method was used for analysis of sucrose in mixed juice, the GC method showed higher precision. The assumption was that the sucrose in the mixed juice inverted due to long run times of automated HPLC analysis.

2.6.2 Brix analysis using refractometers

(Thai and Doherty, 2011) used a refractometer to analyse the brix of the sugarcane juices in their studies involving burnt and green sugarcane extracted from milling processes. According to the SASTA lab manual (2009), juice samples can be analysed for brix using a refractometer. It is recommended that the juice be analysed immediately after they are received in the laboratory or preserved by freezing (especially with juices that have not been heated). Heating under normal processing conditions is said to destroy enzymes and microorganisms, thus reducing the rate of deterioration. Refractometer Brix is defined as the dissolved solids concentration by mass of a sucrose-containing solution obtained using a refractometer (SASTA, 2009) and the measurements are believed to be significantly influenced by the presence of turbidity in the solution, and to obtain results with precision, the turbidity must be removed. Removal of turbidity is best achieved by filtration.

2.6.3 Conductivity ash analysis using a conductivity meter

According to Thai and Doherty (2011), the level of ash correlates with the electrical conductivity (EC) of the juice. The EC depends on the concentrations of ions and their mobility in solution and therefore is associated with the viscosity and brix of sugarcane juice. Studies conducted by Kumar *et al.* (2012) also revealed that the conductivity is linear with the electrolytes and non-sucrose present in sugarcane juice of 20% v/v. The conductivity ash method (SASTA, 2009) gives a measure of the concentration of the ionised soluble salts present in the sample with conductivities of up to 500 mS/cm at concentrations of up to 5 g/100 cm^3. The analytical method of analysing for conductivity ash (SASTA, 2009) is considered applicable for factory juices and involves the use of a conductivity meter. For this method, specific conductivity of a juice at a concentration of 5 g/100 cm^3 or less is determined and compared to the specific conductivity of water. The equivalent ash content of the sample as per convention is calculated by the application of a generic conversion factor.

2.6.4 Colour analysis using a Spectrophotometer

Most researchers and authors mentioned throughout this study who have conducted studies relating to sugarcane juices have resorted to using the ICUMSA (International Commission for Uniform Methods of Sugar Analysis Ltd.) method for analysis of the colour in sugarcane

juices. Studies show that a relationship exists between the absorbance of light through a solution and the colour of a solution and hence a calculation can be used to determine the colour of a sample by measuring the absorbance using a spectrophotometer at the correct wavelengths (Govindaraj and Sankaranarayanan, 1996). According to the SASTA lab manual (2009), the ICUMSA colour method is applicable to all factory juices and is used to determine the ICUMSA colour of the juice at pH 7.00. A diluted sample of juice is filtered through a membrane filter to remove turbidity. The pH of the solution is adjusted to 7.00 ± 0.02 using basic or acidic solutions. The brix and absorbance of the filtered solution is measured at a wavelength of 420 nm using a spectrophotometer and the ICUMSA colour of the solution is calculated. The value of the absorbancy index multiplied by 10 000 is reported as the ICUMSA colour of the solution and the resulting value is expressed in ICUMSA units (IU). Since the wavelength at which the determination of colour in solution is used is set at 420 nm, the value is designated as being the ICUMSA 420 colour.

2.6.5 Near-infrared spectroscopy (NIR) for brix, pol, sucrose, reducing sugars and conductivity ash analysis

NIR has become a popular and useful new analysis method introduced in the sugar industry and is considered applicable to factory juices and can be used for determination of brix, pol, sucrose, reducing sugars and conductivity ash (SASTA, 2009). This alternative to conventional chromatographic methods is said to pose several benefits. According to Perten (2019) NIR provides faster, low cost and easier analyses as compared to the conventional methods of wet chemistry. In addition, the ability to determine multiple parameters without the use of harmful chemicals as in previous methods (such as lead sub-acetate), is desirable to the industry. NIR measures compounds that can absorb infrared light such as water and organic compounds like sugar. However, the limitation is that the concentration levels must not be too low. In many cases, the limit for NIR is approximately 0.1% (Perten, 2019). In cases where concentrations are low, the aforementioned analytical procedures are suggested for each of the respective components.

2.7 Diffuser simulated pilot plant/lab-scale experimental work

This section focuses on the learnings adapted from a review of work on different experimental design approaches and methodologies relating to diffusers that was explored

previously. The purpose of this section was to identify key factors to implement in the design of the pilot extractor and to provide a basis for developing an extraction technique. A considerable number of trials have been performed on pilot plants and laboratory-scale experiments. Attempts have been made to simulate the principles of full-scale counter-current diffusers since they are able to achieve higher extraction rates. Good agreement exists, for almost all of the literature reviewed, that it is challenging to simulate counter-current diffusion on a laboratory-scale experiment because large quantities of sugarcane and numerous pumps are required for each stage in the design. Transportation cost difficulties will exist due to the large size of the extractor and lastly, fibre residence time will become challenging to control since this depends on the bed height and percolation velocity. Studies performed on a pilot-scale previously include packed bed columns, percolation columns, submerged columns, screw conveyor diffusers and batch mixing systems.

2.7.1 Batch mixing systems, submerged and percolation columns

Comparisons between percolation and submerged columns (Buchanan and Jullienne, 1969) showed that the percolation-type diffusers achieve about 5% lower extraction as opposed to the submerged diffuser. This is due to the wetting area and permeability of percolation diffusers reducing the area of the wetted particles and thus the degree of extraction decreases at the equilibrium point. A percolation column is a close approximation to a single-stage diffuser. Such columns allow for percolation rates to continuously be measured. The disadvantages identified in this design includes the high water-to-sugarcane ratios required, the air voids in the bed which hampers extraction as well as the fact that the extraction process is slowed down by percolation. The presence of air in the column reduces the percolation rate. The problem encountered in the study was solved by reversing the juice flow and percolating in an upward direction until the bed was flooded. By doing this, the percolation rate was doubled. The wetting area and permeability of percolation diffusers can be increased by initial upward washing. Channelling in the percolation diffuser is said to reduce the area of wetted particles. Upward flow is suggested to displace air, avoid channelling and improve juice-fibre contact. However, percolation cannot be measured in this case and an alternative column must be used to test percolation rates.

Studies performed on fixed bed columns (Rein, 1972) highlighted that once-through operations prove to be impractical for measuring impurities in the extracted juice as a result

of the juice being very dilute which compromises analytical measurement certainty (Figure 2-10).

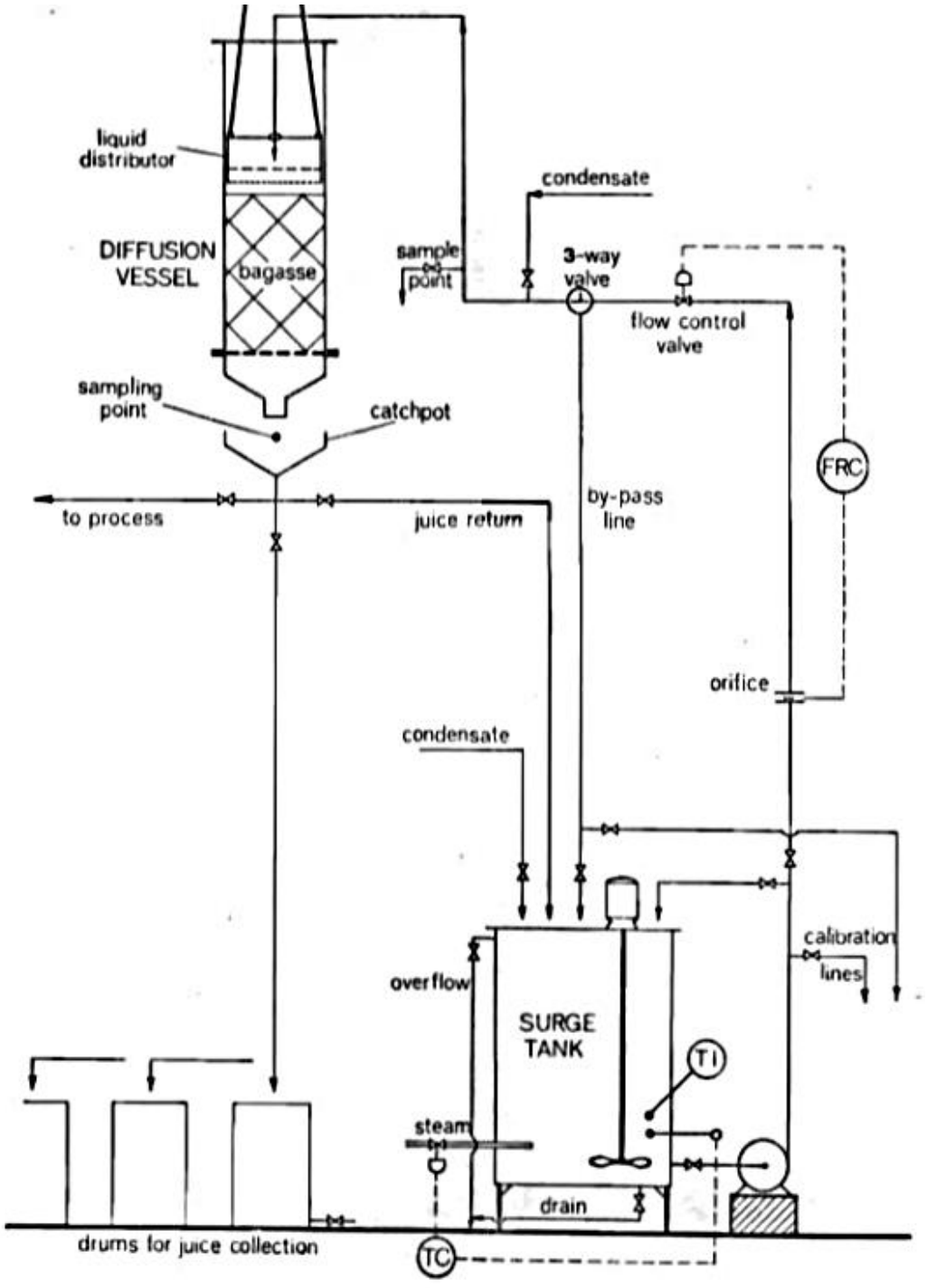

Figure 2-10: Schematic of the pilot plant set-up of a fixed bed column (Rein, 1972)

To prevent such cases, it is suggested that the recirculation of juice be implemented. A further consideration is that the diameter of the column/vessel be selected based on the

sugarcane particle size to avoid wall effects and to ensure that packing densities are similar to those used in full-scale diffusers. However, if up flow is being considered then these factors will not need to be taken into account.

Batch mixing experimental setups studied by Rein (1972) which made use of a rotating paddle (Figure 2-11) and by Lionnet (1985) which makes use of a variable stirrer in the design, promote mass transfer and ensures good mixing and contact between the sugarcane and liquid. Jacketed vessels are introduced in these designs to maintain temperature within the vessel.

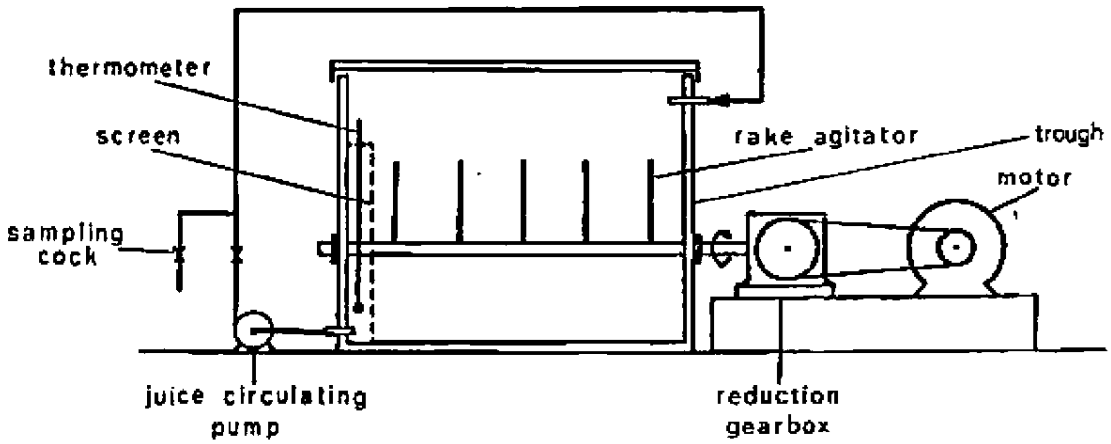

Figure 2-11: Schematic of the lab scale extraction set-up batch mixing system (Rein, 1972)

Studies by Pamu (2012) focused on the effect of different diffusion strategies on sugar extraction from sweet sorghum in a batch and four-stage counter-current experimental set up (Figure 2-12). Attempts were made to replicate the commercial counter-current diffuser process. Results showed that the counter-current process yielded a better extraction than the batch process.

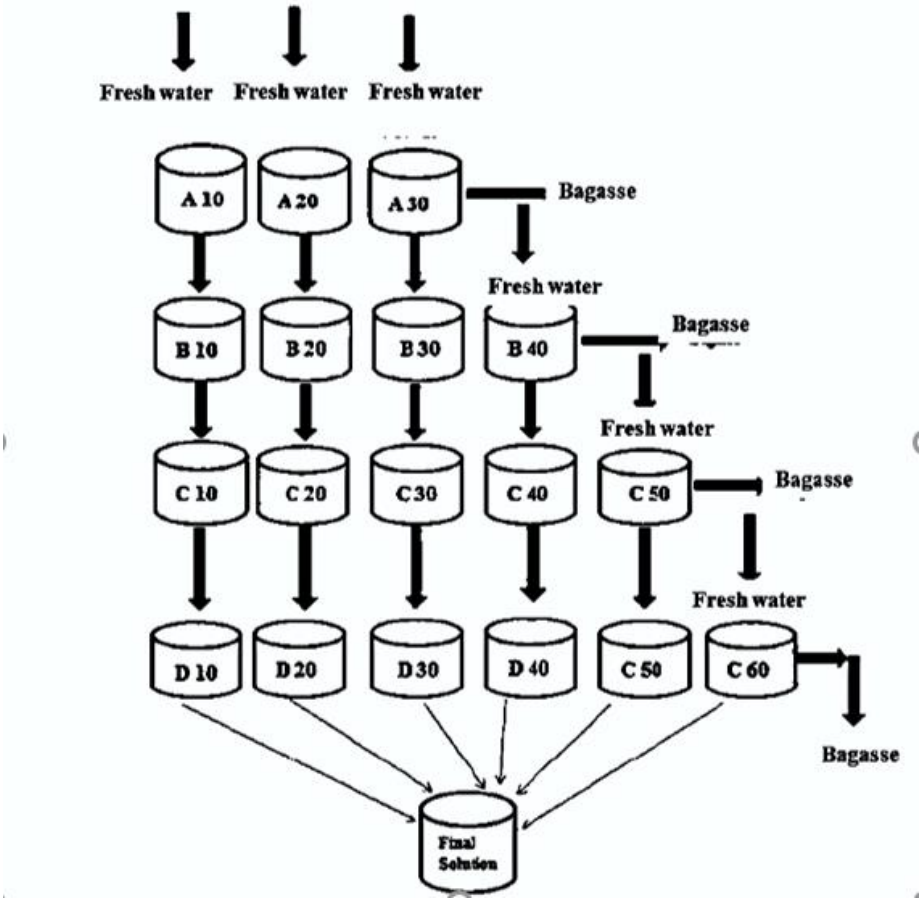

Figure 2-12: Schematic of the four-stage counter-current experimental setup used by Pamu (2012)

2.7.2 Screw type pilot plants

Studies performed on the screw conveyor diffuser highlighted that the counter-current operation achieves the desired concentration gradient for high extraction. It is recommended that the volume of the diffuser be at a minimum to allow for the maximum rate of the liquid passing the sugarcane surfaces. Limitations associated with this setup are that the design is non-ideal for transportation of sugarcane through the diffuser as the sugarcane tends to be dragged with the screw to one side of the trough, which causes channeling of the liquid and thus is not suitable to achieve high extraction (Van der Pol, 1957). In addition, it does not resemble percolation that is generally found in diffusers. Van der Pol (1957) suggested that for such designs, the sugarcane had to be submerged in the liquid at all times to ensure maximum area of contact between the sugarcane and surrounding juice to allow for a concentration gradient. It was also suggested that the volume of the diffuser should be at the minimum to allow for the maximum rate of movement of the liquid past the sugarcane

surfaces. It is suggested that recirculation of both drained and expressed juices would improve recovery from about 87.1% to 96%.

Ming (2007) studied a pilot-scale counter-current extractor (Figure 2-13) to study the extraction of bioactive components using liquorice roots. The effects of temperature, residence time, solvent feed rate and mean particle size of the feed material were investigated. Overall, the extractor unit was ideal in achieving intimate solid-liquid contact which yielded high extraction. Favourable concentration gradients were established. The compression-relaxation effects induced by the rotating screws and counter-current process allowed for better extraction. The screws were said to sometimes assist in squeezing out the liquid from the solid, thereby promoting greater extraction. The effects of temperature and residence time were identified as less significant in the counter-current system due to the intimate solid-liquid contact and the counter-current flow which facilitated the extraction rate.

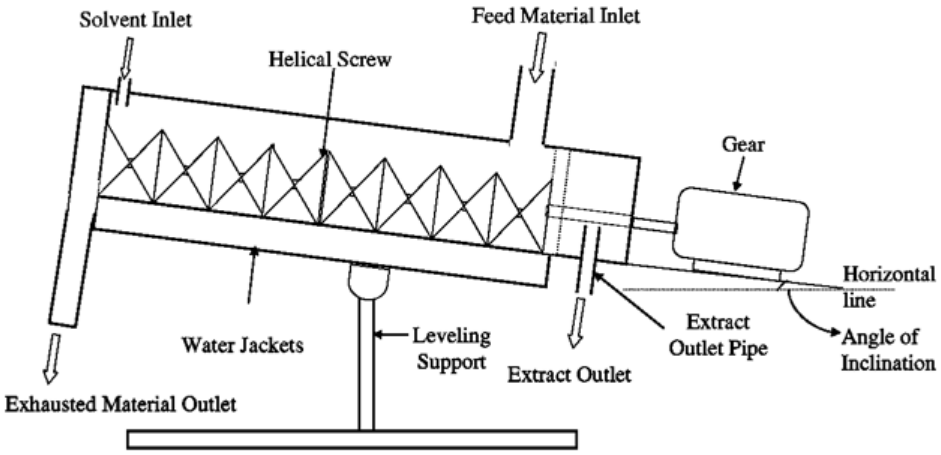

Figure 2-13: Schematic diagram of the counter-current diffuser extraction unit by Ming (2007)

A Limitations identified in the design was that undesirable solid and liquid plug flow can occur due to non-uniform movement. The solids tended to be transmitted faster at the crown of the screw rather than at its bottom. On the other hand, the liquid may not have been percolating through the moving solids bed at a uniform rate due to the non-homogenous permeability of the bed. Finely prepared material tended to settle as a sediment at the bottom of the trough and resulted in plugging of the extract outlet pipe. Excessive disintegration of

solids was identified and was attributed to the shearing force supplied by the screw. For this reason, an extract with more fines from the feed material was observed in comparison to extracts from other extraction methods.

Further general design considerations that resulted from the study included fitting a screen prior to the juice suction pump (Lionnet, 1985) to filter out fibres and thus avoid blockages of the pump and keeping the level of liquid above the sugarcane bed so that the bed is fully submerged in the liquid. Studies by Lionnet (1985) also made recommendations that stainless steel should be used for the construction of the column. Alternative materials of construction present a risk of colour formation believed to occur by the reaction between iron and colour bodies in the sugarcane. In addition, the stainless steel can withstand high temperatures. Lagging should also be considered to minimise temperature losses and a heating jacket (Rein, 1972; Lionnet, 1985) should be used to maintain stable temperature control. Inducing an upward flow of juice will eliminate voidage and will pressurise the juice to enable good solid-liquid contact and thus will promote higher extraction (Buchanan and Julliene, 1969).

2.8 Statistical methods to analyse data

2.8.1 Pearson correlations and coefficients

The Pearson correlation is defined as a measure of a linear association between two variables and the coefficient is usually denoted by *r* (Lund A and Lund M, 2018). The correlation is based on drawing a line of best fit through the data of the two variables. The correlation coefficient is said to be the indication of how far away the data points are to the best fit line. A value of 0 indicates that no association exists between the two variables whereas a value of greater than 0 indicates a positive association and a value less than 0 indicates a negative association. A strong association tends to show an r value closer to +1 or -1. Lund and Lund (2018) gives the following guidelines on how to interpret the values obtained for the correlation coefficient in terms of strength of association; 0.1- 0.3 or -0.1- -0.3 (small), 0.3- 0.5 or -0.3- 0.5 (medium), 0.5-1 or -0.5- -1 (large).

2.8.2 Box and whisker plots

Statistics Canada (2017) describes the box and whisker plot as a graph that presents information from a five-number summary. The graph is said to be a useful tool to establish whether a distribution is skewed and to identify outliers (potential unusual observations). The plots are also useful when large numbers of observations are involved and when two or more data sets are being compared (Statistics Canada, 2017).

2.8.3 Pareto charts

Minitab (2019) recommends a Pareto plot when analysing a factorial design. The plot depicts the absolute values of standardised effects and helps to identify the importance of effects. A reference line is used as a basis for determining which effects are significant. The reference line depends on the significance level and is denoted by alpha (α). Bars that cross the reference line are considered statistically significant.

2.8.4 ANOVA (Analysis of variance)

Statistics How to (2019) describes the ANOVA test as a technique used to find out if experimental results are significant. Groups are tested to establish if there is a significant difference between them. However, the ANOVA test is not capable of establishing which group differs. An LSD (least significant difference) test is performed to go a step further than the ANOVA and enables direct comparisons between two means from two individual groups.

3. EXPERIMENTAL METHODOLOGY

3.1. Viability and verification of the pilot juice extractor

The pilot extractor was designed by a mechanical engineer at the SMRI with considerations from the literature review and then fabricated by an external company. Thereafter the pilot juice extractor was commissioned at the SMRI. This section covers methodologies followed in the commissioning phase and will focus on the following:

- Procedure followed for preparing sugarcane for experiments
- Procedure followed for experiments conducted using the pilot juice extractor
- Procedure followed for experiments conducted using the cold digester
- Procedure followed for experiments conducted using the SASA Cane Testing Service (CTS) press sugarcane sampling procedures
- Sampling procedures for sugarcane and the extracted juices
- Analytical analysis methods followed

The commissioning phase of the project involved the use of the pilot juice extractor, cold digester and press unit. Sugarcane was prepared initially prior to the usage in either of the units and the overview of the experimental procedure followed is shown in
Figure 3-1 for each unit. Detailed operating procedures for the abovementioned units can be sourced from the SMRI.

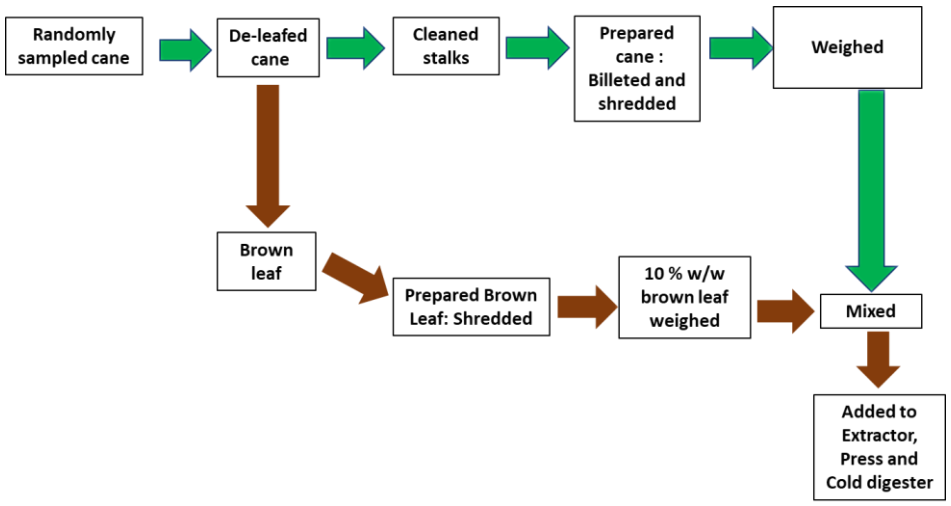

Figure 3-1: Block flow diagram of the sugarcane preparation process during the commissioning phase of project

3.1.1 Sugarcane preparation

Approximately 100 kg of sugarcane was collected from a farm and transported to the SMRI. The stalks were manually topped at the natural breaking point and de-leafed and separated into clean stalks, brown leaves and tops. Each of the contributions were then weighed and the masses were recorded to determine the average percentage contributions of tops and brown leaf in a typical sugarcane variety. The brown leaf was shredded in the Waddell Shredder (Figure 3-2) and stored in buckets to be added later in the trials. For each test, the required amounts of sugarcane were shredded using the Waddell shredder and weighed for the required tests namely extractor (3 kg), cold digestion (2 kg) and press (2 kg) tests. The press and cold digester tests were only performed at random for 6 experiments. Where brown leaf was required, 10% (w/w) of the aforementioned masses of brown leaf was mixed per batch as shown in Figure 3-1 by the brown arrows. For example, in a 1 kg sample, 100 g of brown leaf was mixed with 900 g of shredded sugarcane. The 10% proportion was based on the average amount of brown leaf content determined for two varieties that were used in the tests, namely N12 and N19, which had a brown leaf content of approximately 8.60% and 13.80%, respectively (Table A-1).

Figure 3-2: Waddell shredder used for sugarcane preparation

3.1.2 Pilot extractor methodology

The detailed design and justification for the pilot extractor design choices is described by Loubser (2018). The conceptual design and the fabricated pilot extractor is shown in Figure 3-4. The pilot extractor vessel consists of a stainless-steel removable basket with perforated holes located at the top and bottom screen. A removable basket retainer is designed to keep the basket in place at the top whilst a Teflon support ring is located at the bottom of the column to secure the basket in place. A vortex breaker is installed in the column to avoid /prevent a vortex from forming at the top of the column.

The extractor has two heating media available: a steam jacket and an electric heating element. Instrumentation on the pilot plant includes a temperature probe, conductivity meter, an in-line brix refractometer and a magnetic flow meter. The system includes a manual globe valve to alter the flowrate, a ball valve to drain out a sample or excess water and a solenoid valve to control the steam supply. The extractor is attached to a control box with a main power supply and a programmable logic controller (PLC) for data logging, trend display and control via a user interface, by which the pump and heat supply can be controlled.

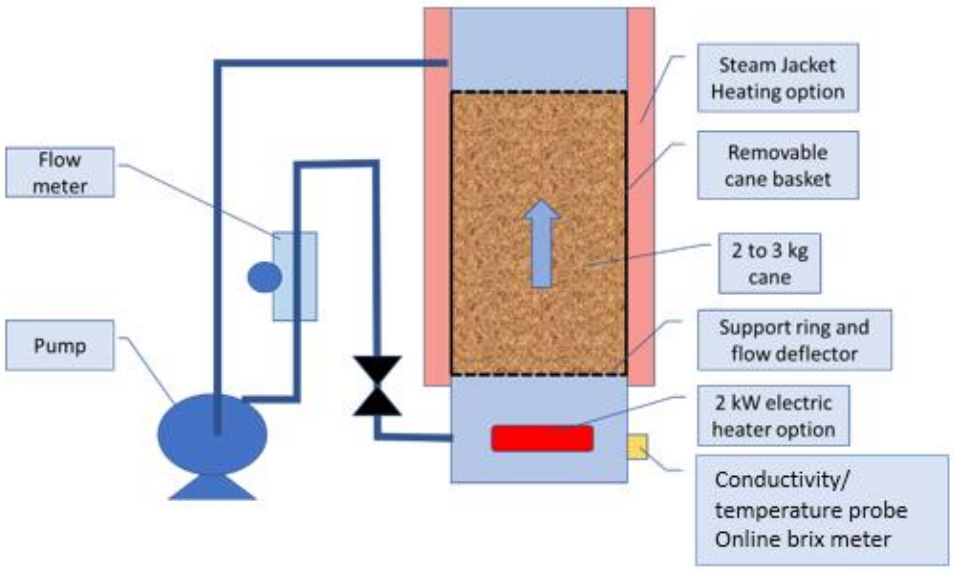

Figure 3-3: Conceptual design of the pilot juice extractor (Loubser, 2018)

Figure 3-4: The pilot juice extractor as built

The pilot extractor was operated as follows for the commissioning phase:

- The column (without the basket) was filled with hot water (approximately 60°C) to a level of 7 cm above the outlet of the column. The water was recirculated whilst heating with steam until the set point of 90°C (approximately 10°C above the experimental set point) was reached.
- The basket was packed with 3 kg of shredded sugarcane.
- Once the temperature set point was achieved, the column was drained of a set quantity of water and the basket was inserted into the column.
- The temperature set point was adjusted to the desired value of 75, 80 or 85°C and the main pump, brix pump, steam/electrical heating and data logging was started.
- The flowrate was monitored and controlled by manually adjusting the globe valve to achieve the required 45 L/min set point.
- The recirculation was continued for 30, 45 or 60 minutes at the temperature set point.
- The juice was collected in a bucket per test from the drain valve after the extraction process was completed.
- The juice in the bucket was mixed with a stirrer.

- Approximately 500ml of juice sample was taken and transferred to five 100 ml sachets for freezing and analysis.
- Following sampling, the basket with spent sugarcane was removed, the sugarcane discarded, and the column rinsed with clean water twice.
- Approximately six pilot extractor runs were conducted per day.

The tests carried out for commissioning are presented in

Table ***3-1***. A full standard operating procedure for the pilot extractor can be sourced from the SMRI.

Table 3-1: Experiments carried out for the extractor tests

Test number	Brown leaf	Time (minutes)	Temperature (^{o} C)
15	No	30	85
5	No	45	80
16	Yes	60	85
9	No	30	85
3	Yes	30	75
12	Yes	45	80
13	No	30	85
14	No	60	75
8	No	60	75
1	Yes	30	75
11	No	45	80
6	Yes	45	80
17	No	45	80
10	Yes	60	85
2	No	60	75
7	Yes	30	75
18	Yes	45	80
4	Yes	60	85

3.1.3 Press methodology (Figure 3-5)

A two kg shredded sugarcane sample was used for the pressing test. The press cylinder was lowered and compacted the sugarcane to a pressure of 200 MPa, squeezing juice out of the sugarcane which flowed through small perforations in the basket into a saucer. The juice was collected from the saucer and analysed. The press juice sample (approximately 500 ml) was collected in a bucket, mixed well and transferred into a Schott bottle before being transferred to the 100 ml sample sachets for freezing and analysis. The Schott bottle was rinsed out with a portion of press juice. A detailed standard operating procedure of the press can be sourced from the SMRI.

Figure 3-5: The press unit and its control system at the factory

3.1.4 Cold digester /DAC methodology

A two-kilogram sample of the prepared sugarcane batch was taken for the DAC test. 1000 g of shredded sugarcane was placed in the digester. Two kilograms of cold water was added to the digester. The tests occurred at room temperature for a period of 20 minutes. The DAC juice sample was decanted through a screen and approximately 500 ml was collected, mixed well and transferred into a Schott bottle before being transferred to the 100 ml sample sachets. The Schott bottle was rinsed out with a portion of DAC juice. The sachets were

stored in the cooler box. A detailed standard operating procedure of the cold digester can be sourced from the SMRI or SASTA lab manual (2009).

3.1.5 Analysis of juices

The juices extracted from the pilot extractor, cold digester and press were analysed for ICUMSA colour (SMRI, 2013), reducing sugars by HPLC measurement (SMRI, 2011), brix using a handheld refractometer (SMRI, 2018c), sucrose (SMRI, 2014) and conductivity ash (SMRI, 2014).

3.2 Validation

Prior to the validation trials, the green sugarcane project team conducted a lithium tracer test to determine the diffuser residence time in order to sample the draft juice accurately and correlate it to the consignment of sugarcane used in the other techniques. The project team also conducted a mock one-day sampling trial. This section presents the methodology followed for:

- The one-day sampling observation and tracer test
- Sugarcane sampling procedures at the factory
- Procedure followed for draft juice sampling from the commercial diffuser at the factory
- The pilot juice extractor, cold digestion and CTS press tests performed at the factory

3.2.1 One-day sampling trial/observation at Gledhow factory

A one-day observation was carried out at the Gledhow Sugar Mill to understand the process from the weighbridge to the diffuser line. The purpose of the observation was to identify any bottlenecks or challenges that may have arisen during validation and to use the information to establish a smooth operating plan for the green sugarcane validation trials. The observation showed the following actions occurring on-site in the areas of interest.

- Incoming sugarcane was weighed at the weighbridge. A delivery slip provided by the truck number indicated the sugarcane variety as well as the truck number which was located on the side of the truck and used to identify the truck.
- The truck was then directed to stand in line at the diffuser line to load the sugarcane on the spiller (either at the diffuser or milling tandem) whichever requires sugarcane first.
- At the tracking station, the tracker identifies on the screen (when the sugarcane is loaded on the spiller) if the sugarcane will be tested. At this point, a sample number was allocated. The sugarcane could thereafter be tracked using the sample number.
- At the diffuser, a screen showed the samples being tracked and indicated when the CTS sampling flap would open.
- The amount of sugarcane to be sampled at this point could be altered by the sampler.
- Sugarcane that was not being tested by CTS could still be tracked as a special sample.

3.2.2 Lithium tracer test at Gledhow factory

The methodology used to conduct the lithium tracer test was according to Davis (1996). Approximately 8.5 kg of lithium chloride was dissolved in 12.08 kg of water to deliver the required lithium concentration of 6.75 % (m/m). A shredded sugarcane sample was collected from the CTS sampling hatch and was soaked in the lithium chloride solution for a period of 15 minutes. Following the soaking period, the sample was dumped onto the fresh shredded sugarcane conveyor just ahead of the CTS sampling point all at once and the draft juice was sampled at the draft juice pump every 30 seconds for 15 minutes, then every minute up to 30 minutes and thereafter sampled every 2 minutes until the total time of 5 times the calculated diffuser residence time was reached (approximately 100 minutes). More details are presented in Appendix B.

3.2.3 Diffuser layout at Gledhow Sugar Mill

The Gledhow Sugar Mill diffuser (BMA model) is a 12-stage counter-current diffuser. The diffuser has a design throughput of 120 tons of sugarcane per hour (Wienese, 2002). The length of the diffuser is approximately 52.3 m while the width is approximately 4 m. The screen area is approximately 209 m^2. According to Barker and Davis (2010), the chain speed for the diffuser can vary between 0.65 m/min and 0.90 m/min, with an average of

0.75 m/min. The average fibre residence time is about 70 minutes (Barker and Davis, 2010). The volume of each juice compartment is about 3.3 m^3 and the larger scalding juice draft juice compartment is about 18 m^3 to give a total juice volume of 60.9 m^3. There is one scalding juice heater which heats the juice to 92 - 95°C with V1 steam (vapour bleed from the evaporator station). There is direct steam injection with V1 steam in the front juice compartment and stages 2, 4, 6, 8, 10 and 12. A sump pump located under the diffuser, pumps spilled juice to the press water swirl tank which is then returned to the diffuser just before the second set of lifting screws. The throughput is 88 t/hr which is 73% of the design throughput of 120 t/hr. The extraction to date was 97.19% and there is no mud recycle.

3.2.4 Procedures for sampling at the factory

Sugarcane sample (Figure 3-6): Only sugarcane consignments above 30 tonnes in mass were selected for the validation tests to ensure the diffuser was filled with enough sugarcane so that an accurate sample of draft juice could be taken. The sugarcane truck was identified by SMRI personnel at the weighbridge and the details of the delivery note were recorded. The details included the sugarcane variety, grower name, tonnage, time of cutting and the truck identity number and whether the sugarcane was burnt or unburnt. The truck driver was then directed to wait in line to load the diffuser spiller for the selected cane.

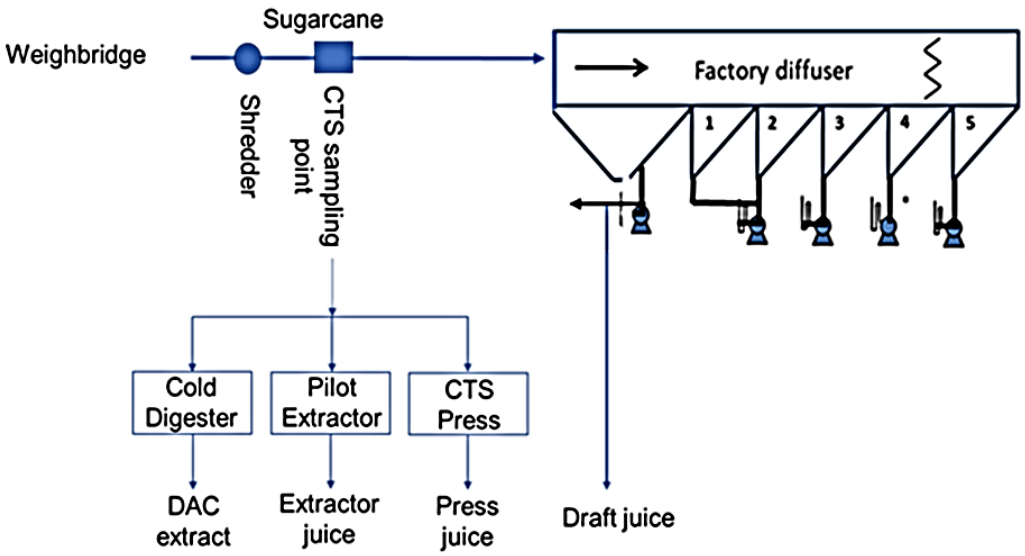

Figure 3-6: Flow diagram showing sampling of shredded sugarcane and sugar juices extracted for the validation phase

The SMRI personnel then made their way to the tracker to retrieve the sample number of the sugarcane consignment in order to track it along the CTS sampling point. A computer screen then indicates the path of the sample of cane as it moves to the preparation phase and reaches the diffuser. Once the CTS sugarcane sample point was reached, an 8-9 kg sample of shredded sugarcane was taken from the CTS sampling hatch in two 25 litre buckets with the assistance of the CTS cane sampler. The shredded sugarcane sample was placed on a plastic sheet, mixed well, sampled in even proportions, weighed for each test and further sub-sampled into three samples. Each sub-sample was directed to the pilot extractor, the CTS press or cold digestion (DAC) for further processing to extract the juice. A total of 16 consignments of sugarcane were tested.

Draft juice sample (Figure 3-7 and Figure 3-8): When the tail of the sugarcane consignment passed the CTS sampling point and sampling had ceased, a stopwatch timer was started to determine the time when the draft juice had to be sampled. Once the timer reaches 8-9 minutes (determined by the lithium tracer test), the draft juice sample was taken from the draft juice sampling point at the sugarcane diffuser by opening the gate valve. The bucket was rinsed once with the draft juice and the sample was taken and transferred (approximately 500 ml) into a Schott bottle and taken to the lab for cooling and storing into 100 ml sachets.

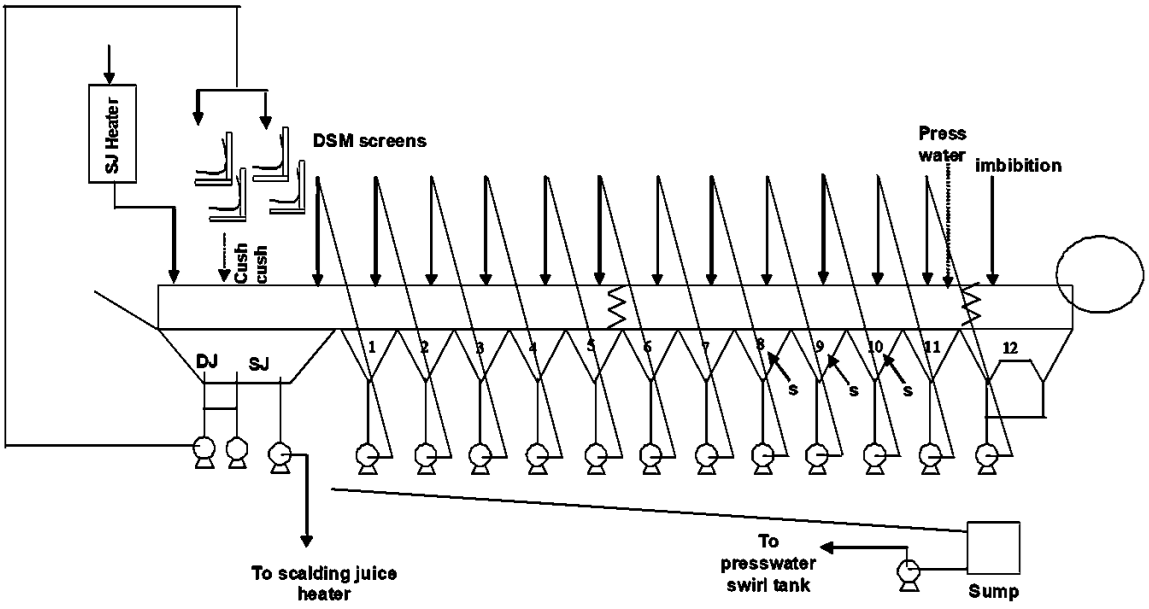

Figure 3-7: Schematic of the Gledhow diffuser (Barker and Davis, 2010)

Figure 3-8: The sugarcane diffuser at the Gledhow factory showing the stages

DAC extract sample: 500 ml sample was retrieved from CTS labs and cooled and stored in 100 ml sachets. Samples were then frozen with dry ice and transported to the SMRI. The procedure followed for cold digestion can be sourced from the SASTA lab manual (2009). The press juice sample was retrieved as per procedure followed during commissioning.

The extractor methodology was carried out following the same procedure as the commissioning tests with a few differences to be noted. The steam supply at the factory used was V1 steam (Figure 3-9) which was tapped off and directed to a point near the extractor. Steam was throttled and maintained manually during the experiment using the gate valve according to temperature readings that were logged on the computer interface during the experiment. The solenoid valve used in the commissioning tests could not be used since it seemed to be getting stuck with the use of V1 steam. The temperature set point was adjusted to the desired value of 80°C. The recirculation was continued for 30 minutes at the temperature set point. Three pilot extractor runs could be achieved per day.

Figure 3-9: The area where the extractor was placed at the factory with the red arrow showing the V1 tap off steam supply and gate valve.

All juices obtained from the respective methods above were analysed at the SMRI labs using suitable methods as mentioned in Table 3-2.

Table 3-2: Summary of analysis performed on the juices obtained from various extraction methods

Analyte	Diffuser /Draft juice	Pilot extractor juice	Press juice	DAC extract
ICUMSA Colour (SMRI, 2013)	✓	✓	✓	✓
Brix (SMRI, 2018c)- Wet chemistry		✓	✓	✓
Sucrose (SMRI, 2018b)- Wet chemistry		✓		
Conductivity ash (SMRI, 2018a)- Wet chemistry		✓	✓	✓
Fructose and glucose (SMRI, 2018b)- Wet chemistry	✓	✓	✓	✓
SMRI-NIRS brix (SMRI, 2014)	✓		✓	✓
SMRI-NIRS sucrose (SMRI, 2014)	✓			
SMRI-NIRS conductivity ash (SMRI, 2014)	✓			

3.3 Brown leaf trials

3.3.1 Sugarcane preparation methodology

The cleaned sugarcane stalks required for the tests were approximately 125 kg per variety of green sugarcane and 40 kg per variety for burnt sugarcane. Each variety was collected and subsequently cleaned (topped and de-leafed) (Figure 3-10). The mass of resulting leaves, tops and cleaned sugarcane stalks (Figure 3-11) were determined per variety and the percentage contributions of each calculated thereafter. The contributions per variety is shown in Appendix C: Table C-1. Details of the sugarcane harvesting, burning, collection and testing dates can be viewed in Appendix C: Table C-2.

Figure 3-10:Photograph of topping and de-leafing green sugarcane

Figure 3-11: Photographs of the resulting tops, brown leaf and cleaned sugarcane stalks after the cleaning process of green sugarcane

For each test, the cleaned sugarcane stalks were chopped, weighed and prepared (shredded) using the Waddell shredder. For tests requiring the addition of brown leaf, the required mass of brown leaf (either 7.5% or 15% m/m representing medium and high levels respectively) was added to the cleaned and chopped sugarcane stalks and then shredded together in the Waddell shredder. The prepared sugarcane sample was then mixed well, subsampled and then directed to the pilot extractor, press, cold digester for DAC analysis (when required) and the percolation column. The prepared sugarcane was also used for moisture analysis and sugarcane density tests.

Since the trial was to be conducted over 10 days, DAC control tests were performed on all four varieties to measure the quality of the sugarcane at the beginning of the trial to assist in determining whether significant sugarcane deterioration took place during the tests. All DAC control tests were done on green sugarcane without brown leaf. The results from these tests are shown in Appendix C: Table C-6.

3.3.2 Pilot extractor method, cold digester and press method

The same methodology was followed for the pilot juice extractor as mentioned for the validation phase and the cold digester and press method as mentioned for the commissioning phase. Approximately six tests could be achieved per day. Full standard operating procedures can be sourced from the SMRI.

3.3.3 Percolation column methodology

- The percolation tests were conducted simultaneously as the pilot juice extractor tests
- Approximately 6 kg of shredded sugarcane with or without brown leaf were sprinkled in the percolation column (Figure 3-12)
- Water was filled in the column and heated to a temperature of 60 °C using electrical heaters
- The percolation column pump was switched on once the desired temperature of the water was reached and the pump percolated water through the sugarcane bed for a period of 30 minutes.
- The sugarcane flowrate programme logged the data for each experiment

- More specific details on the standard operating procedure can be sourced from the SMRI

Figure 3-12: Percolation column filled with shredded sugarcane and in operation

3.3.4 Sugarcane density tests

- Approximately 1.2 kg of shredded sugarcane with or without brown leaf was used for the sugarcane density tests
- The sugarcane density apparatus (Figure 3-13) was used for the tests and consists of a housing column and a plunger column
- The required amount of shredded sugarcane was sprinkled into the housing
- 1.5 kg of water was weighed in a beaker
- The plunger was placed in the housing and the water from the beaker was transferred into the plunger
- A timer is started for 20 seconds and immediately after the height of the sugarcane was measured using three rulers located on the column (points on each of the rulers was read and recorded)
- More details on the standard operating procedure can be sourced from the SMRI

Figure 3-13: Sugarcane density apparatus

3.3.5 Moisture analysis

- Moisture analysis was performed in triplicate
- Three trays were initially tared and labelled
- Approximately 300 g (100 g per tray) of prepared sugarcane with or without brown leaf was used
- The trays were placed in the Heavy-duty lab oven (at 105°C) for drying overnight
- The mass of the dried sample was then recorded, and the % moisture calculated thereafter by difference of masses before and after.

3.3.6 Analysis of juices

The juices extracted from the pilot extractor were analysed for ICUMSA colour, reducing sugars by HPLC measurement, brix and sucrose and conductivity ash. The DAC extract was also analysed for the same components using the abovementioned methods with the exception that brix and sucrose was determined by the SMRI NIR method. These methods are discussed in detail in section 2.6 and 3.4.

3.4 Analytical methods

This section presents the methodology followed for each analysis performed on the extracted juices, either during the commissioning, validation or brown leaf phases.

All juices from the selected extraction procedures were subsampled and transferred into five 100 ml plastic sachets and frozen using dry ice. The frozen samples were transported to the SMRI for storage in a freezer and analysis for the various analytes later on by the analytical laboratory at the SMRI. The analytes for which each of the juice extracts was analysed for commissioning are mentioned with each of the methodologies described earlier. All juices extracted by each method was considered mixed juices and were analysed accordingly due to the juice characteristics being very similar to a mixed juice. The final results were all standardised and reported on brix. No dilution of juices was required for each method since the brix was low for all methods.

3.4.1 Procedure followed for Brix using wet chemistry (SMRI, 2018c)

The juice sample was filtered using a Celite 577 filter aid and the auto filter using Whatman 6 filter paper. The sample was then read on the refractometer at a temperature of 20°C. Juices are analysed immediately after thawing to avoid deterioration. The tolerance associated with the Brix analysis is ± 0.05°Bx. More details on the method are available in the SASTA lab manual (2009).

3.4.2 Procedure followed for sucrose using wet chemistry (SMRI, 2018b)

The juice sample was filtered using a Celite 577 filter aid and the auto filter using Whatman 9 filter paper. The method uses gas chromatography with flame ionisation detection (FID) and an internal standard (trehalose dihydrate) to determine sucrose contents of a sample quantitatively. Results are based on the peak area to mass ratios of an integrated chromatograph. The sugars in the filtered juice sample are volatilized by silylation using a trimethysilyl (TMS) reagent. The TMS-ethers of the various sugars are separated from each other and other volatile components using a low to medium polarity column. The relative standard deviation (RSD) for sucrose should be less than 1% for duplicate analyses. Details on the method followed as well as the calculations is available in the SASTA lab manual (2009).

3.4.3 Procedure followed for ICUMSA colour using wet chemistry (SMRI, 2013)

All juice samples were filtered using a membrane filter (0.45µm pore size) via vacuum filtration. The brix of the juice is measured using a refractometer as per the standard method. The pH of the solution is adjusted to 7.00 ± 0.02 using acidic/basic solutions (hydrochloric acid - 0.05 M) or sodium hydroxide - 0.05 M). The samples (20°C) were read on a spectrophotometer using a 5 mm cell at a wavelength of 420 nm. The result is reported as absorbance and is fed into a calculation to determine the ICUMSA 420 colour in ICUMSA units (IU). More details on the method and calculation followed can be sourced from the SMRI or in the SASTA lab manual (2009).

3.4.4 Procedure followed for conductivity ash using wet chemistry (SMRI, 2018a)

Samples were filtered using the Celite 577 filter aid and the auto filter using Whatman 6 filter paper. Samples were read at 20°C using a conductivity meter. The results were reported in micro siemens (µS) and are used in a calculation to determine the conductivity ash. The cell constant of the conductivity meter was determined via a calculation using the reading obtained from a 0.0025 M KCl solution. The brix was also determined using the refractometer as per the standard method. More details on the method and calculation can be sourced from the SMRI or in the SASTA lab manual (2009).

3.4.5 Procedure followed for SMRI-NIR for Brix, Sucrose, Conductivity ash (SMRI, 2014)

All juices were filtered using Celite 577 and the auto-filter using Whatman 6 filter paper. The samples were then read on the NIR using the mixed juice equation. More details on the method can be sourced from the SMRI.

3.4.6 Procedure followed for fructose and glucose using HPLC with PAD (SMRI, 2011)

2.5 g of filtered mixed juice and 10 g lactose solution (preparation detailed in SMRI (2014) weighed in triplicate in 50 cm^3 volumetric flasks and made to the mark. 2 cm^3 (using PAD-2 cell) or 1 cm^3 (using PAD - SC cell) were diluted to a 100 cm^3 solution with 0.02% azide solution (preparation detailed in SMRI (2014)) in a second volumetric flask. An injection

volume of 20 µl is used for all samples. Three standards (preparation detailed in SMRI (2014) are run initially to determine the linearity of the system and corrected before reading all samples. The full method is available from the SMRI.

4. RESULTS AND DISCUSSIONS

4.1 Viability and verification of the pilot juice extractor

The main objectives of the tests were:

- To determine if the extractor could differentiate between juice extracted from sugarcane with and without brown leaf.
- To define suitable operating conditions (time and temperature specifically) for the extractor to be operated at the sugar factory during the validation phase.
- To conduct a comparison between two established sugarcane methods namely the cold digestion (DAC -Direct analysis of cane) and press method.

The start of the commissioning tests introduced two design changes that had to be implemented before further work. A Teflon basket retainer was added as well as a vortex breaker to prevent overflow and reduce channelling. Furthermore, the tests highlighted bottlenecks in the general procedure of the extractor tests, generated a basis for the operating procedure and allowed for rectification of any glitches in the unit design.

4.1.1 Design of experiments

The TIBCO Statistica TM (version 13.4.0.14) software was used to develop a two factorial experimental design for the combinations of operating conditions. The three independent variables selected for the design of experiments were identified from the review of literature. Temperature, retention time and the addition of brown leaf were selected as the three independent variables. Sugarcane preparation was considered a blocking factor due to the limitation of the Waddell shredder preparing the sugarcane at only one setting. It was decided that temperature and contact time be tested at two levels 75°C and 85°C and 30 and 60 minutes respectively. A centre point of time at 45 minutes and a temperature of 80°C was selected. The brown leaf addition variable was tested at two levels (with and without brown leaf). Brown leaf was added at 10% on weight of sugarcane. An average value of brown leaf content for two sugarcane varieties (high and low colour) was used to determine the brown leaf weighting (Appendix A: Table A-1). Literature was used to determine the aforementioned temperature and contact time experimental levels. It was agreed that the responses (dependent factors) to measure the quality of the juices would be sucrose, reducing

sugars (Fructose and Glucose), conductivity ash and ICUMSA colour using their respective analytical instrumentation and techniques. One sugarcane variety (high colour – N12) was tested due to the limiting factor of time in order to proceed with validation at the factory prior to their shut down for the season. Statistica yielded a result of 18 tests (Appendix A: Table A-2) to be carried out for the commissioning phase, including two repeats per condition. Tests were randomised by the software and carried out accordingly. Randomisation allowed for the reduction of confounding effects that would otherwise bias the results.

The tests were performed for each of the 18 experiments and the raw data for the pilot extractor juice that was collected is presented in Appendix A: Table A-3. Sucrose, reducing sugars and conductivity ash were calculated and reported on g per 100 g brix to standardise the results for all methods since the pilot extractor juice, DAC extract and press juices vary in brix concentrations.

4.1.2 Comparison of average juice quality extracted from sugarcane with and without brown leaf

The experimental design for the pilot juice extractor allowed for a comparison of the average juice quality (of three samples) to be determined at one set of conditions in the presence and absence of brown leaf at 80°C for 45 minutes (Figure 4-1).

For both brown leaf and no brown leaf conductivity ash and reducing sugars, there were large variations in results with large standard deviations. The large standard deviations may be attributed to very low concentrations present in the extractor juice together with low brix concentrations, which points towards large analytical relative uncertainties rather than the pilot extractor method not being repeatable. A marked difference was observed for colour and gravity purity for juices extracted with and without brown leaf and these analytes did not display standard deviations that were as large as with the other analytes mentioned previously (Figure 4-1).

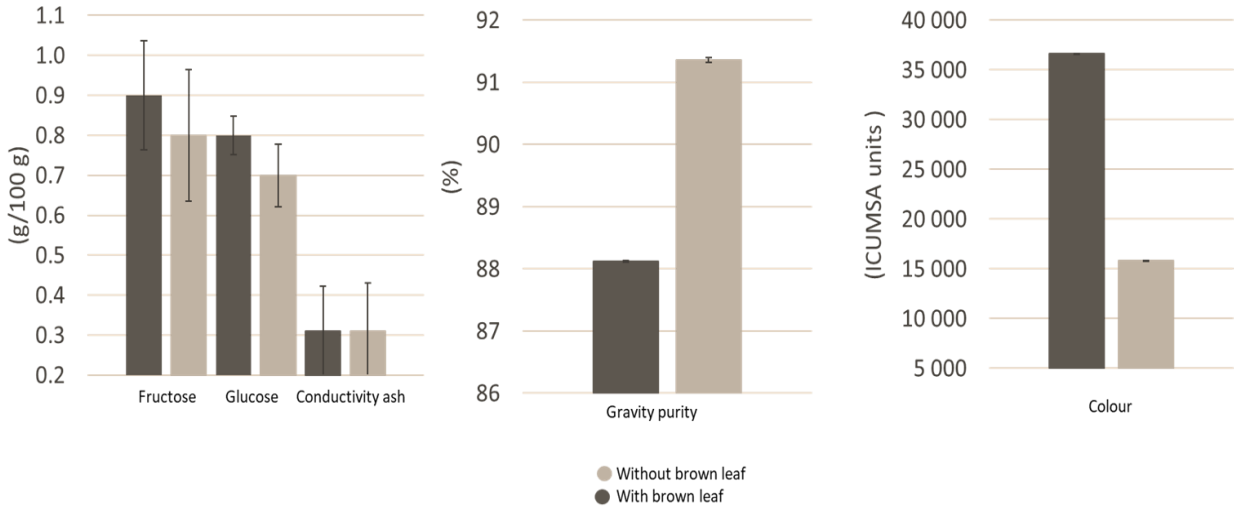

Figure 4-1: Comparison of average juice results from the pilot extractor tests in the presence and absence of brown leaf at 80 °C for 45 minutes

4.1.3 Statistical significance of variables

4.1.3.1 Pilot juice extractor assessment of juice extracted from sugarcane with and without brown leaf

Table 4-1 presents a quick summary of the significance or otherwise the effects of the three independent factors (leaves, time and temperature) on the response variables (colour, gravity purity, fructose and glucose concentrations and conductivity ash- measuring the quality of juice). The statistical tests were performed at a 95% confidence interval. Time had a statistically significant effect on reducing sugars (fructose and glucose) only. Temperature had no statistically significant effect on any of the dependent variables. Figure 4-2 to Figure 4-7 presents the results in more detail.

Table 4-1: Statistical evaluation of the effects of the variables on the analytes tested (p values > 0.05)

Variables	ICUMSA colour	Gravity purity	Fructose	Glucose	Conductivity ash	RS/ash ratio
Brown leaf	✓	X	✓	✓	X	X
Time	X	X	✓	✓	X	X
Temperature	X	X	X	X	X	X

Key: RS = Reducing sugars
✓ = statistically significant
X = not statistically significant

A Pareto chart of standardised effects was used to determine significant effects with a 95% confidence interval (p = 0.05) for which all bars crossing the red line were statistically significant. Overall the results revealed that the addition of brown leaf had the most significant effect, whereas temperature and retention time were significant for minimal responses only. It is hypothesised that the ranges tested for these variables were too narrow to see marked effects and that the dependence of measured values on the time and temperature in the ranges tested were smaller than variances in the method and sampling. However, temperatures outside of the range tested would not be representative of normal diffuser operations and therefore it was decided to operate the pilot extractor at 80 °C in all subsequent tests. In addition, it was noted that the addition of brown leaf used in all the experiments occurred in a manner such that separate brown leaf quantities were shredded each time and used per experiment rather than shredded as one large quantity. This could have caused variations in the results since tests showed that there exists a variation in the nature of brown leaf.

The result obtained for the effect of all the factors tested on the response of gravity purity are shown in Figure 4-2. None of the factors tested were found to be statistically significant. The result is questionable since purity should be affected by time and temperature as well as the addition of brown leaf. The result therefore points to the reasoning that the range tested for these factors was too narrow to have shown marked effects.

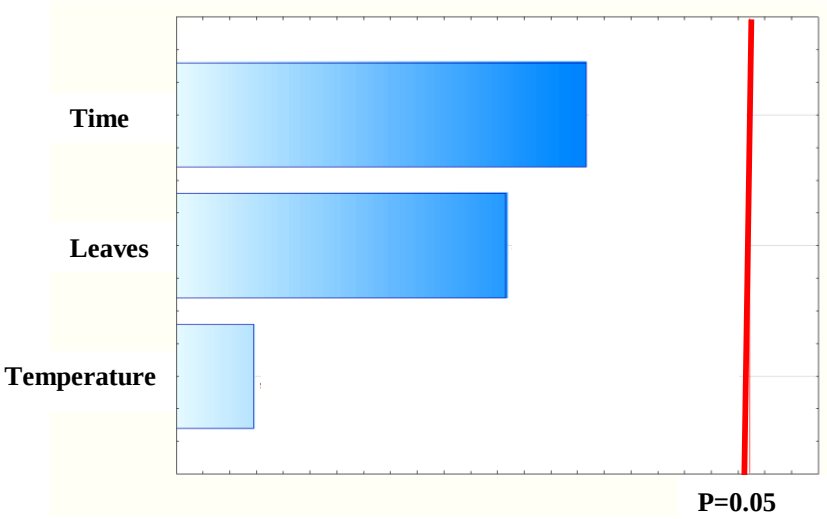

Figure 4-2: Effects of temperature, time and brown leaf on gravity purity %

The addition of brown leaves showed a marked effect on ICUMSA colour whereas time and temperature showed no significant effects (Figure 4-3). The result associated with the presence of brown leaf is expected, since brown leaf is believed by its characteristics in nature to add colour, which is in accordance to the literature reviewed (Lionnet, 1985). Higher temperatures at longer contact times should cause an increase in colour (Lionnet, 1985) however, this was not observed in this case and can be attributed once again to the narrow ranges tested.

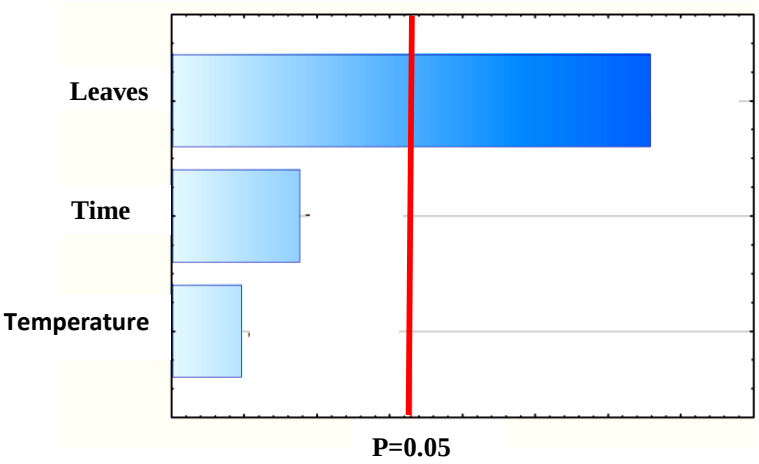

Figure 4-3:Effects of temperature, time and brown leaf on ICUMSA colour

The addition of brown leaves showed the significant effects on glucose and was very close to the line for fructose (Figure 4-4 and Figure 4-5), whereas temperature showed no statistically significant effect. The contact time showed an unexpected result at a first glance where it displayed a statistical significance but a negative effect indicating that the reducing sugar levels decreased over time. After careful study, it was hypothesised that the fructose

and glucose results were reported on brix and it is assumed that the fructose and glucose may have exited in the first 30 minutes whilst brix continued to increase over extended times, resulting in a decrease in the ratios causing the effect that was observed. Rein (1972) saw a similar effect and confirms that in a once-through system, reducing sugars are extracted more rapidly than sucrose. Very high temperatures should ideally have significant effects on glucose and fructose levels (Graham *et al*., 1968), but the results showed that temperatures tested (75°C, 80°C and 85°C) were possibly out of range in this case for the pilot extractor process which is different to other pilot units that were tested in previous studies.

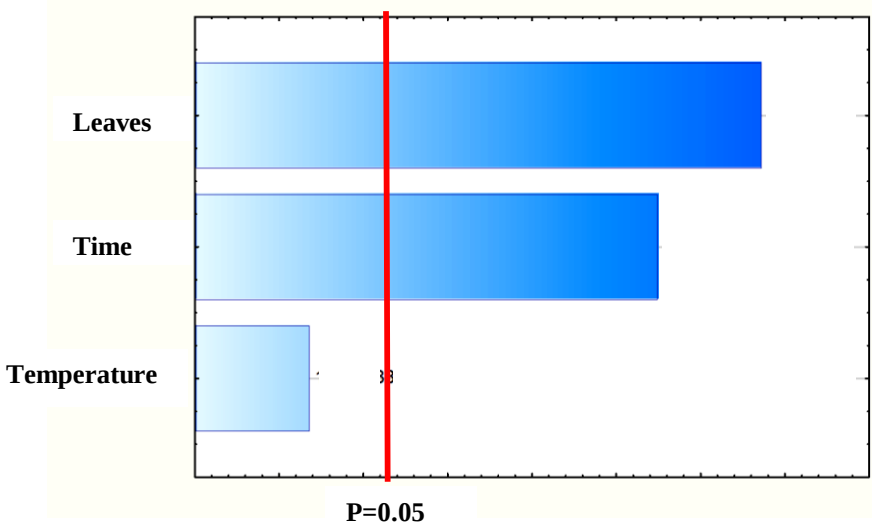

Figure 4-4: Effects of temperature, time and brown leaf on glucose (g/100g brix)

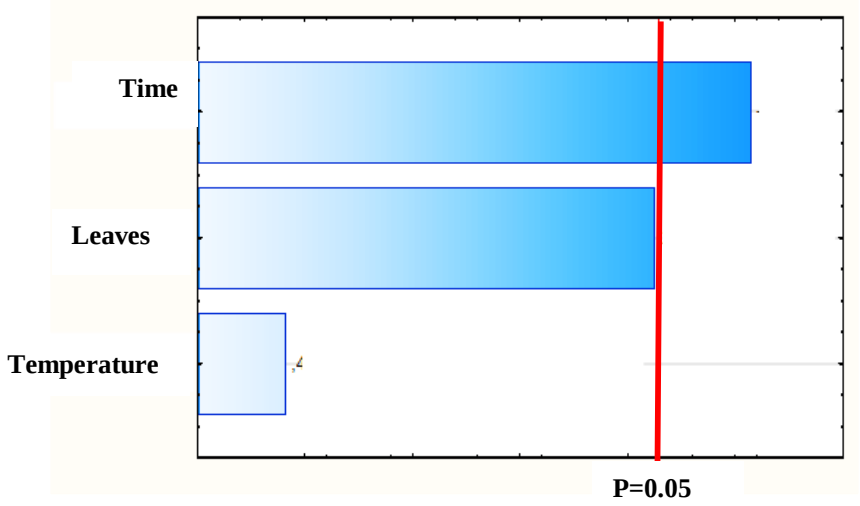

Figure 4-5:Effects of temperature, time and brown leaf on fructose (g/100g brix)

The addition of brown leaves, temperature and time showed no significant effect on conductivity ash (Figure 4-6). According to Rein (2007), increases in ash content are expected with addition of brown leaf, due to the nature of the brown leaf. It was of concern that the SMRI NIR methods (SMRI, 2014) used for the analysis of conductivity ash in the juices were not suitable since it was later established that the equation developed for mixed juice which is used for this method would not accurately represent the extractor juice since the matrix of the extractor juice is very different. At this stage, suggestions were made to look at an alternative methods of analysis of the extracted juice for future work.

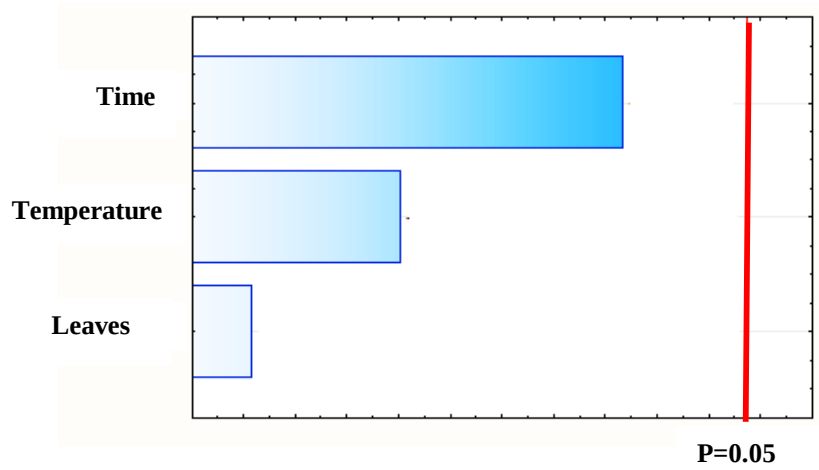

Figure 4-6:Effects of temperature, time and brown leaf on conductivity ash (g/100g brix)

No significant effects were observed for the reducing sugar: ash ratio (Figure 4-7). The result was expected due to the results observed with the reducing sugars and conductivity ash.

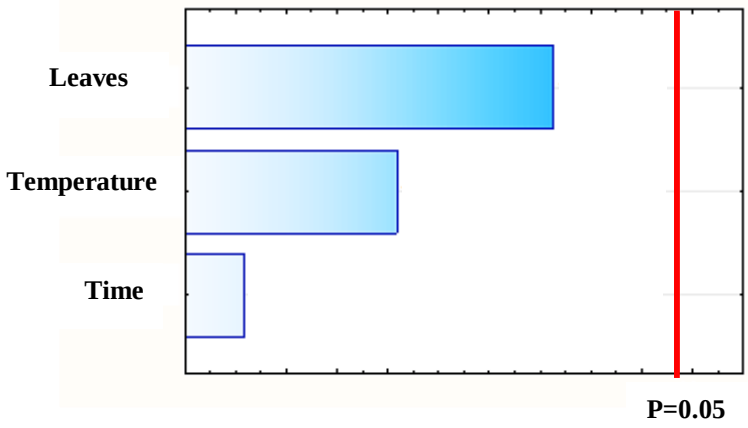

Figure 4-7:Effect of temperature, time and brown leaf on reducing sugar ash ratio

4.1.3.2 Operating conditions for the pilot juice extractor

The results for the determination of suitable operating conditions (objective 2) indicated that an operating time of 30 minutes would be suitable to enable more tests per day since no significant differences in juice quality were observed by increasing the time to 45 or 60 minutes. Similarly, a temperature of 80°C, similar to the average diffuser temperature in a factory and previous studies conducted, was suggested for use in the validation trials since no significant differences were observed for the ranges tested. It is hypothesised that the ranges tested for both parameters as guided by literature may have been too narrow to have seen significant differences.

4.1.3.3 Comparison of the pilot juice extractor against other established methods

The final objective of this phase was for the pilot extractor juice to be compared to the DAC extract and press juice as a basis for determining if the juice extracted from the pilot extractor was not the same quality as the juice extracted from the two existing methods. Experiments were conducted and results are reported for an average of three random tests (representing each of the six different experimental conditions (3 with and 3 without brown leaf) for the cold digester and the press methods. The pilot juice extractor results (Appendix A: Table A- 3) are reported on an average of 6 tests (comprised of three tests with no brown and three tests with brown leaf) for the range of processing conditions. The cold digester and press results are reported in Appendix A: Table A-4. In addition, consideration had to be given, when interpreting the results, that the pilot extractor temperature and time were varied whereas for cold digester and press, the temperatures and times remained constant for all tests. It was hypothesised that the extractor results would lie between the DAC and press method since the DAC method is believed to extract the maximum from the sugarcane including impurities whereas the press method is believed to extract the minimum due to its simple mechanical expression of juice at room temperature (Barker and Davis, 2013).

For gravity purity with and without the inclusion of brown leaf (Figure 4-8), the extractor juice result was between the DAC extract result and the press juice result with the press juice displaying the highest purity and the DAC extract, the lowest purity. The result was expected, since the cold digestion method is believed to extract the maximum (Buchanan, 1967), including the impurities, which would result in a lower overall juice purity. The press method extracts the minimum and therefore may not contain as many of the extracted

impurities. The graphs also indicate that the presence of brown leaf introduces more impurities in the juice since the purity for juices extracted from sugarcane with the presence of brown leaf extracted by all methods is lower than the juices extracted from sugarcane without brown leaf.

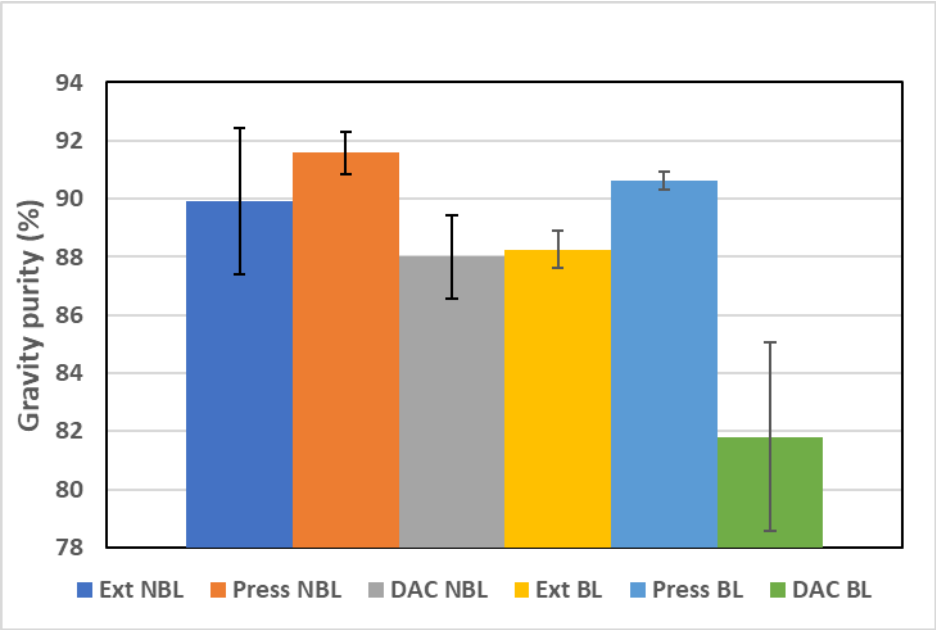

Figure 4-8:Gravity purity of juices extracted from sugarcane with and without brown leaf using the pilot juice extractor (Ext), cold digester (DAC) and CTS press (press) methods. NBL (No brown leaf) and BL (Brown leaf)

The colour results (Figure 4-9) observed when comparing the three extraction methods without brown leaf illustrated that the pilot extractor juice showed the highest colour content whereas the press juice showed the lowest colour content from the three methods. The result may be due to the higher temperatures and extended retention times (longer exposure with higher temperatures) used in the pilot extractor method as compared to the cold digester method which is believed to form colour. The press juice result was expected. With the inclusion of brown leaf the DAC extract displayed the highest colour content, which was expected.

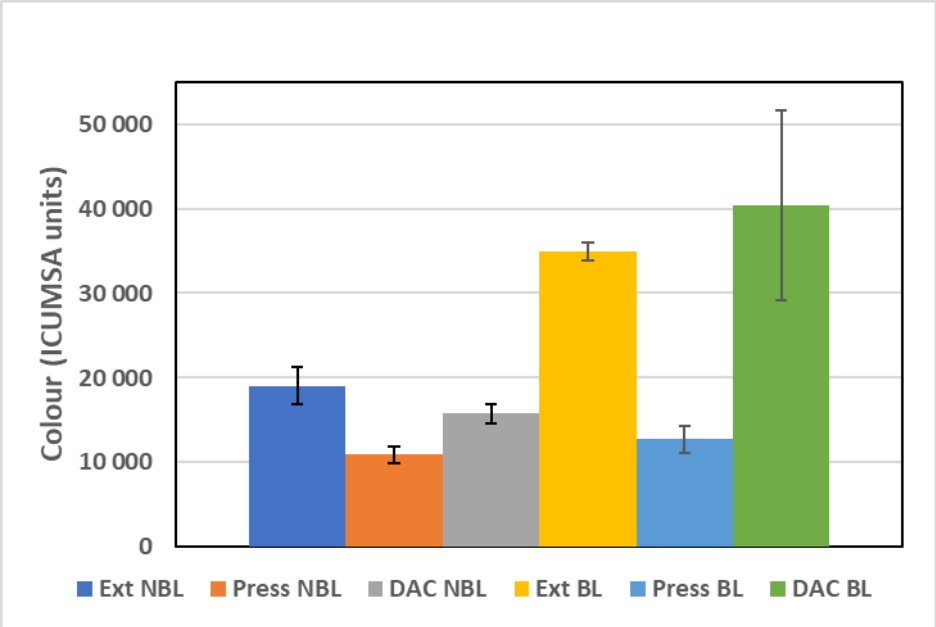

Figure 4-9: Colours of juices extracted from sugarcane with and without brown leaf using the pilot juice extractor (Ext), cold digester (DAC) and CTS press (press) methods. NBL (No brown leaf) and BL (Brown leaf)

The fructose and glucose results (Figure 4-10 and Figure 4-11) for juices extracted from sugarcane with and without brown leaf, suggest that the press and extractor juices almost overlap, whereas the DAC extract displays a higher fructose and glucose concentration of more than twice the amount. The result is expected since the cold digestion method should by its nature, extract most of the fructose in comparison to the pilot extractor and press method. However, it was expected that the pilot extractor juice should have displayed much higher concentrations of the reducing sugars compared to the press, due to the exposure of higher temperatures. It is hypothesised that the concentrations of these components present in the extractor juice could have been underestimated due to analytical error associated with the low brix of the juices extracted from the pilot extractor.

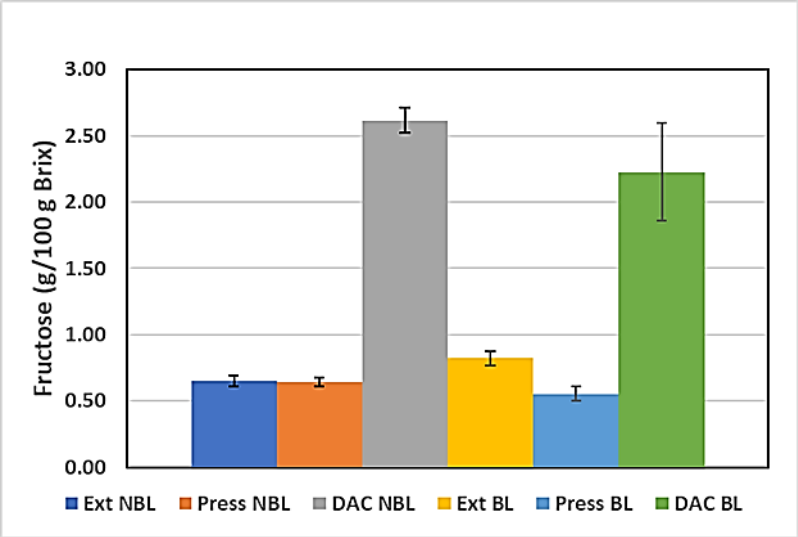

Figure 4-10:Fructose content (g/100 g Brix) in juices extracted from sugarcane with and without brown leaf using the pilot juice extractor (Ext), cold digester (DAC) and CTS press (press) methods. NBL (No brown leaf) and BL (Brown leaf)

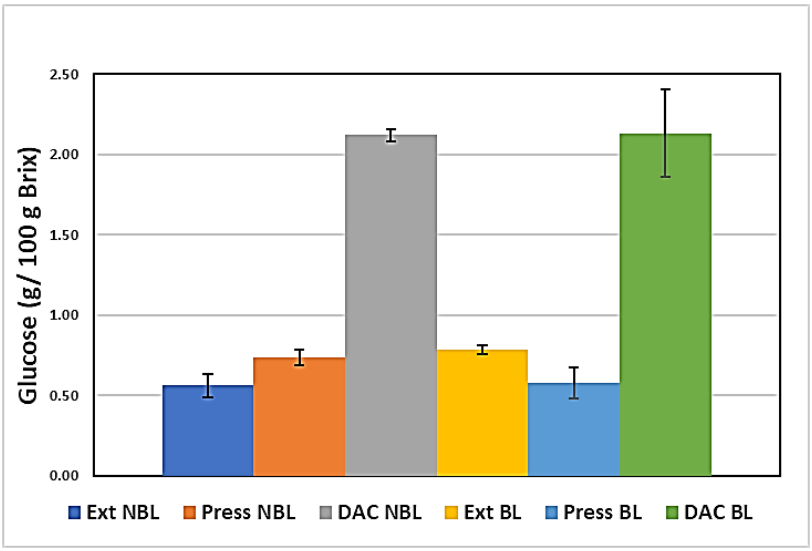

Figure 4-11:Glucose content (g/100 g Brix) in juices extracted from sugarcane with and without brown leaf using the pilot juice extractor (Ext), cold digester (DAC) and CTS press (press) methods. NBL (No brown leaf) and BL (Brown leaf)

The results observed for the conductivity ash for juices extracted from sugarcane with and without brown leaf (Figure 4-13) showed minimal differences considering the standard error. However, it can be deduced that a slightly higher conductivity ash concentration is observed

overall for juices extracted by the press and extractor methods, with addition of brown leaf, which is expected since the brown leaf is said to contain high levels of ash, as discussed in the literature review. The DAC results showed minimal differences when considering the standard error, which suggests that it may have also been slightly higher in this case. The question of whether the NIR analytical method was suitable for analysing the low brix pilot extractor juice was raised by the green cane steering committee since the equations were originally developed for a mixed juice of a much higher brix and a different matrix. It was decided from here onwards that for the validation tests, wet chemistry methods be used to analyse conductivity ash concentrations.

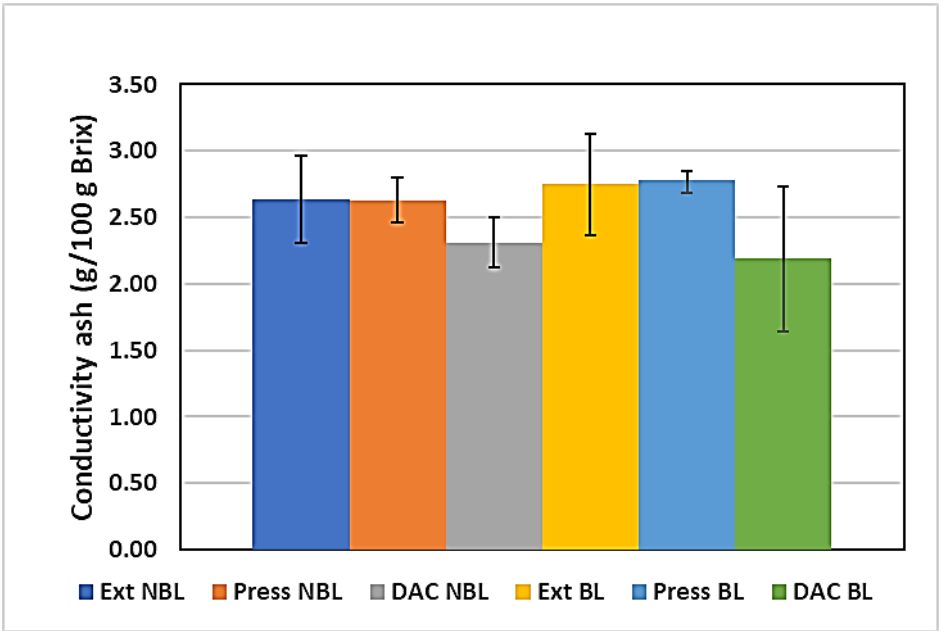

Figure 4-12:Conductivity ash (g/100 g Brix) in juices extracted from sugarcane with and without brown leaf using the pilot juice extractor (Ext), cold digester (DAC) and CTS press (press) methods. NBL (No brown leaf) and BL (Brown leaf)

The first objective of the commissioning phase was achieved by showing that the pilot juice extractor method produced a juice in which significant differences between burnt and green sugarcane (more specifically, with and without brown leaf) could be measured. The second objective, recommending operating conditions of time and temperature for the pilot extractor to be run during the validation trials was also met, indicating that a time of 30 minutes and a temperature of 80°C would be suitable. The third objective, comparing the pilot juice extractor with the two established methods (cold digestion and pressing) in terms of juice

extraction for sugarcane with and without brown leaf was also achieved. Overall results showed that the press, cold digestion and pilot juice extractor method are significantly different from each other and that the pilot extractor juice quality parameters are likely to lie between the two established methods for most of the juice content parameters that were measured.

4.2 Validation trials

The validation phase objectives aimed to:

- Compare the juice extracted from the pilot extractor against the juice extracted from a full-scale diffuser for different sugarcane varieties.
- Compare the juice extracted from the cold digestion, press and pilot extractor method against the draft juice and determine which method is the best representation of the draft juice quality.

During validation, 16 sugarcane consignments were tested (four green sugarcane and 12 burnt sugarcane samples). The details of the sugarcane consignments tested are shown in Appendix A: Table A-1. Only four samples of green sugarcane were tested as the Gledhow Sugarmill preferred sending green sugarcane to the milling tandem as opposed to the diffuser, since the cane preparation on the diffuser line is not optimised for green sugarcane and the high brown leaf content tend to cause chokes. It was decided that the green sugarcane data points be omitted for the determination of the correlations since there is a fundamental reason to believe that the draft juice from these consignments was not in itself representative of a draft juice from a diffuser line optimised for green sugarcane. However, the purpose of this study was not to validate the pilot extractor against a commercial diffuser, specifically for green sugarcane samples but rather to test whether the pilot extractor was able to produce an extract from which the equivalent draft juice from an appropriately set up diffuser might be inferred. No significant differences in juice quality were observed for draft juice obtained from sugarcane extracted in the presence (green sugarcane) and absence (burnt sugarcane) of brown leaf. Some differences in juice quality were observed for juice extracted from green and burnt sugarcane by the other extraction methods, which supports the suggestion that non-sucrose from the leaf associated with green sugarcane was not well extracted in the

diffuser or that mixing in the diffuser diluted the effects. However, the green sugarcane data points are still shown in the scatterplots as open points.

The lithium tracer test performed on the GH diffuser front-end assisted with the determination of the draft juice sampling time for the validation trials and the methodology and the results of the test are detailed in Appendix B.

4.2.1 Correlation determination using scatterplots and statistical interpretation of the results

This section presents the results for colour, gravity purity, glucose, fructose, conductivity ash and non-sucrose for the juice extracted from the pilot juice extractor, cold digester and press respectively, compared with draft juice from the commercial diffuser. The scatterplots present the trend lines and correlations, whereas the box and whisker plots present the statistical comparisons. The extractor juice gravity purity measurements were lower than press juice purity measurements and higher than that of the DAC extract measurements. The pilot extractor juice purity measurements was also closer to the draft juice measurements than the juice extracted by the other two methods (Figure 4-13).

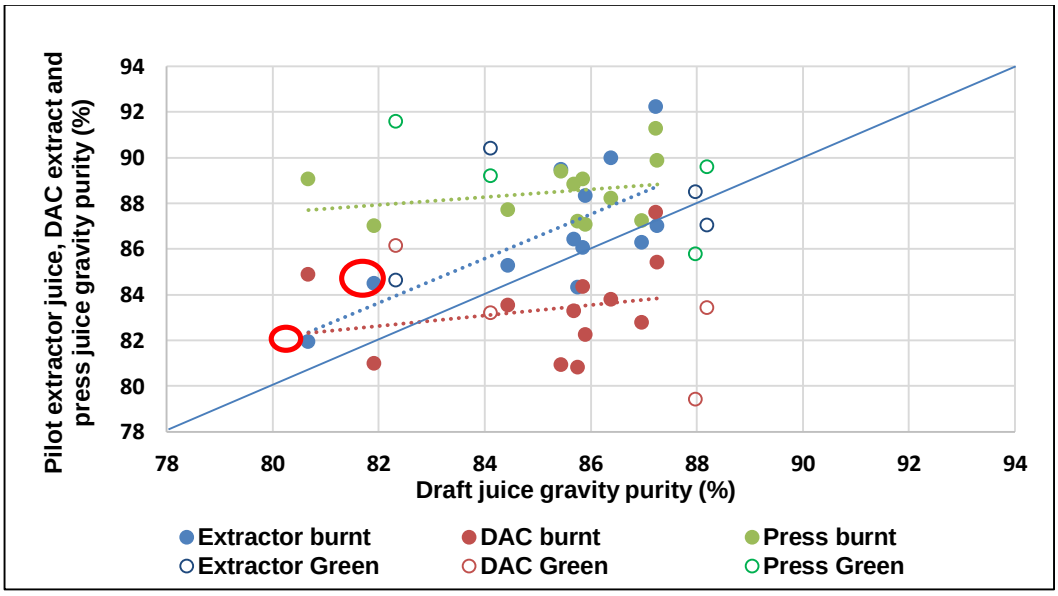

Figure 4-13: Gravity purity correlations for extracted juices against draft juice with the equivalence line represented by a solid blue line

Larger variations in measurements were observed for green sugarcane across all extraction methods when compared to burnt sugarcane (Figure 4-14) and it is evident that the green sugarcane points showed a lot of scatter across all methods (Figure 4-13). The trend line shows a slight bias with the purity of the pilot extractor juices higher than that of the draft juices. The samples circled in red were highlighted as they were sampled after factory stops and this may have affected the quality of the draft juice due to deterioration of the juice.

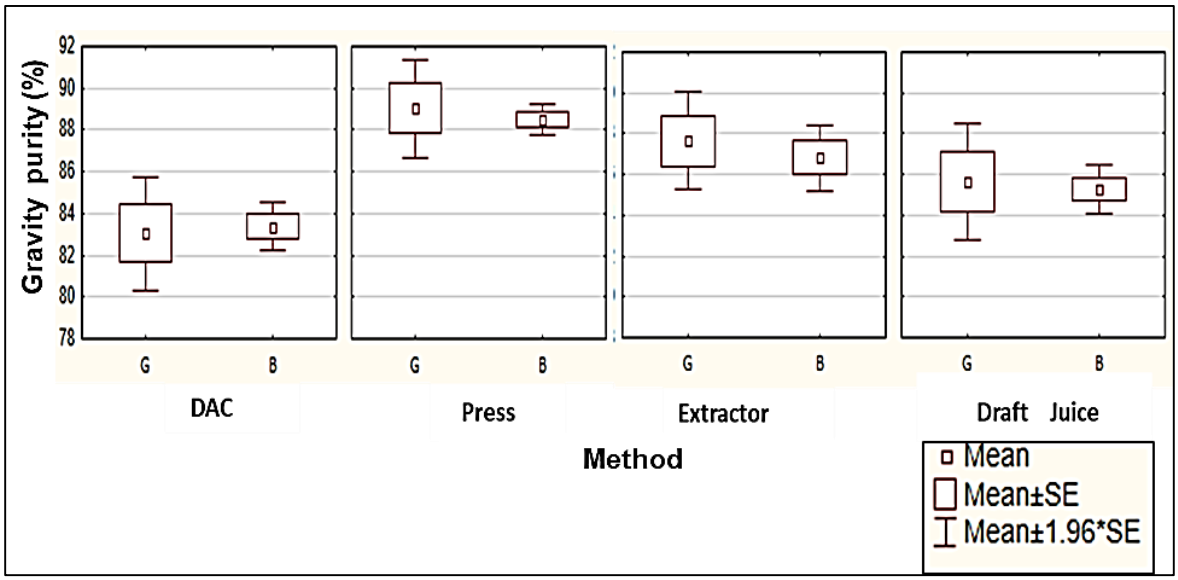

Figure 4-14: Box and whisker plot showing the comparison of cold digestion, press, pilot extractor and draft juices for green and burnt sugarcane for gravity purity. G - green sugarcane, B – burnt sugarcane, SE – standard error

The trend lines representing the colour content in juice from the pilot juice extractor were closer to the equivalence line than the trend line representing the colour content in juice from the cold digester and press methods (Figure 4-15). The colours of extractor juice measurements were lower than those of the DAC extracts and higher than those of press juices (Figure 4-15).

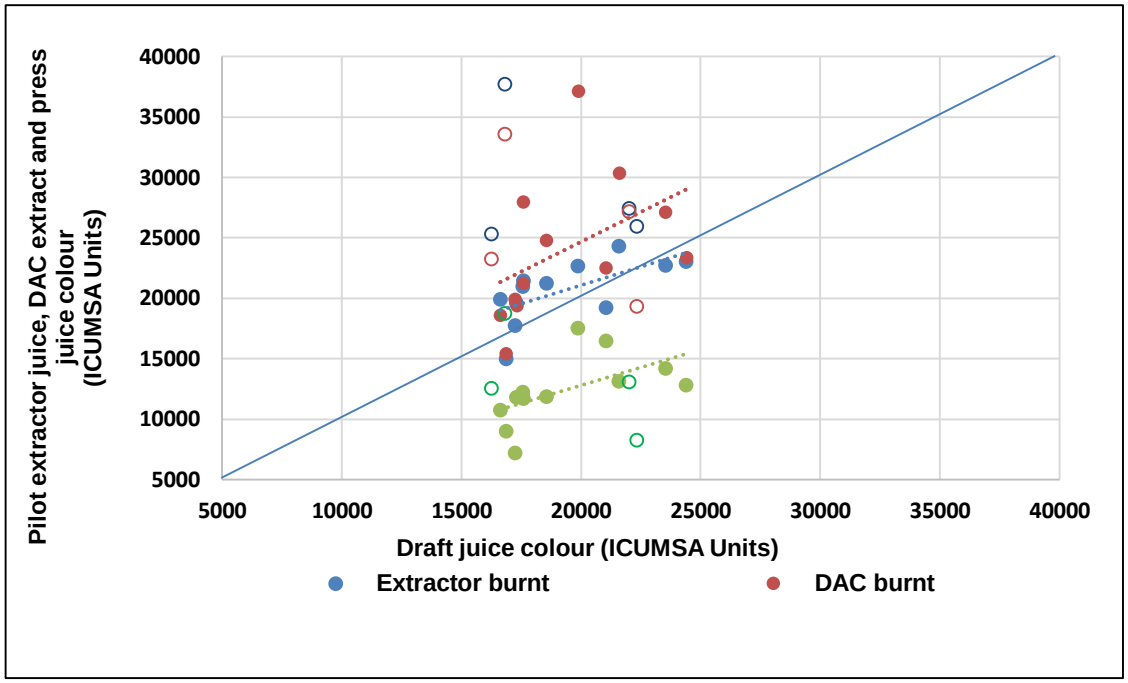

Figure 4-15: ICUMSA colour correlations for extracted juices against draft juice with the equivalence line represented by a solid blue line

From the three extraction methods, the colours of the juices from the pilot extractor were closest to those of the draft juices. Minimal differences in colour in draft juice, DAC extract and press juice extracted from green and burnt sugarcane were observed (Figure 4-16). The only significant difference in colour was observed for juices extracted from burnt and green sugarcane by the pilot extractor (Figure 4-16). The extraction of colour from leaves is typically a slow process. The leaves tend to move with the fibre and are therefore subjected to the full residence time in the diffuser (usually long enough to extract colour). The colour will exit towards the tail-end of the diffuser and only after some time will it exit in the draft juice. This is most likely to have occurred after the draft juice was sampled at nine to 10 minutes in the validation study.

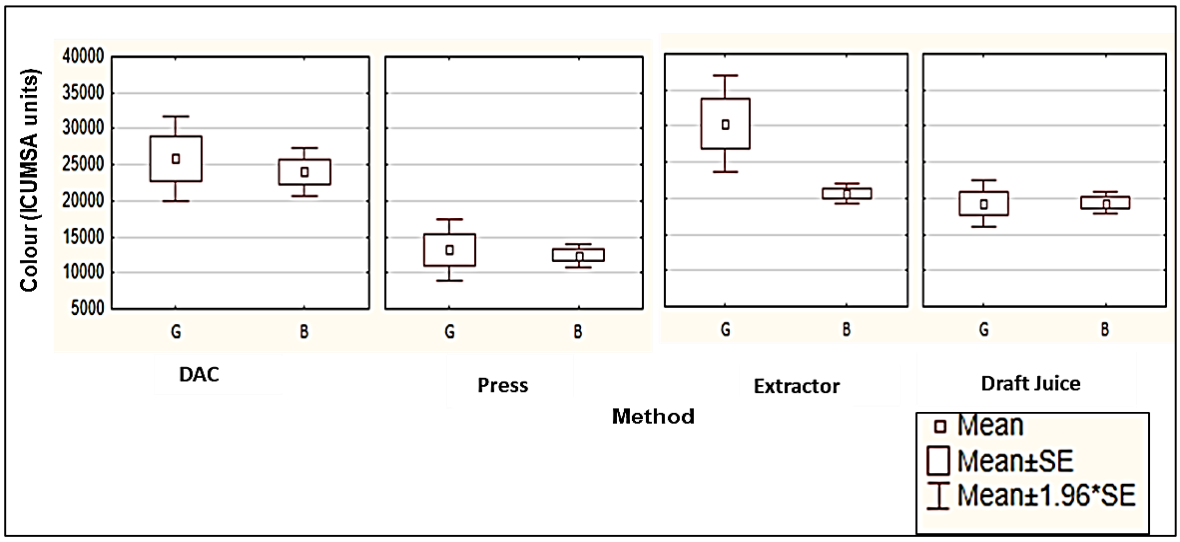

Figure 4-16: Box and whisker plot showing the comparison of cold digestion, press, pilot extractor and draft juices for green and burnt sugarcane for ICUMSA colour. G - green sugarcane, B – burnt sugarcane, SE – standard error

The trend lines for the glucose content in juice from the pilot juice extractor was closer to the equivalence line than the trend line for the glucose content in juice from the cold digester and press methods (Figure 4-17). The glucose content measurements for the pilot extractor juice was closer to the draft juice measurements than those of the press juice or DAC extract measurements (Figure 4-17). For both types of reducing sugars, draft juice showed a very large variability (Figure 4-18) and consideration must also be given to the fact that reducing sugars are present in low concentrations in all juices and it is possible that analytical uncertainties are relatively high. These factors may have contributed to a deviation in the expected results.

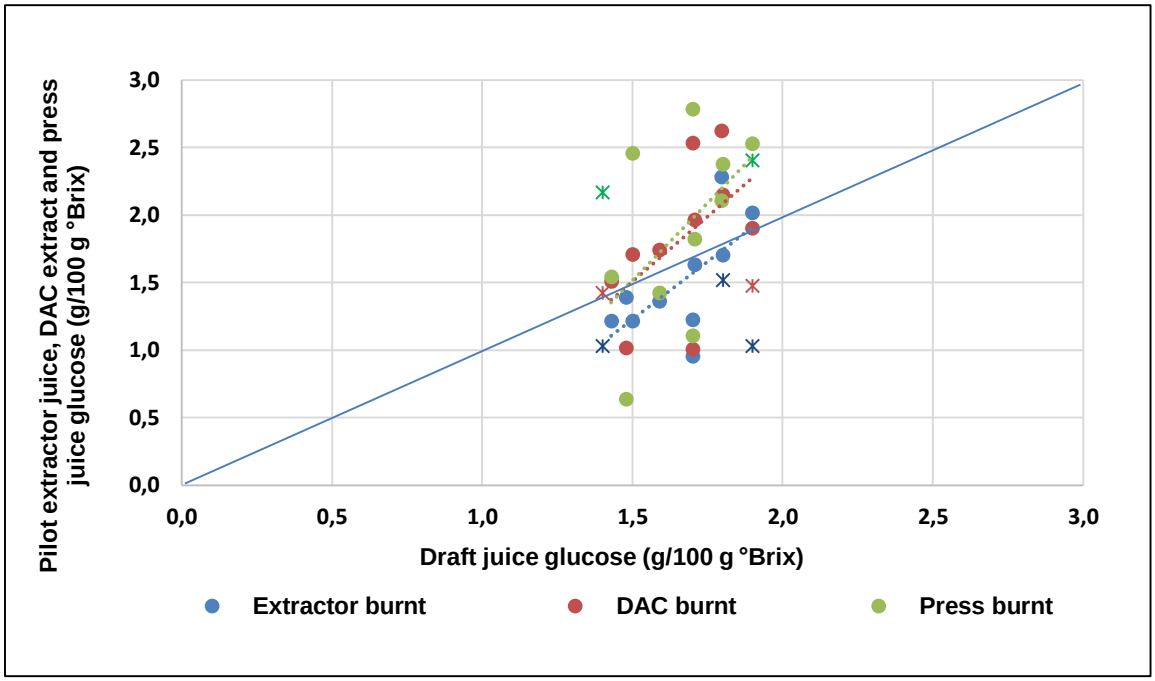

Figure 4-17:Glucose correlations for extracted juices against draft juice with the equivalence line represented by a solid blue line

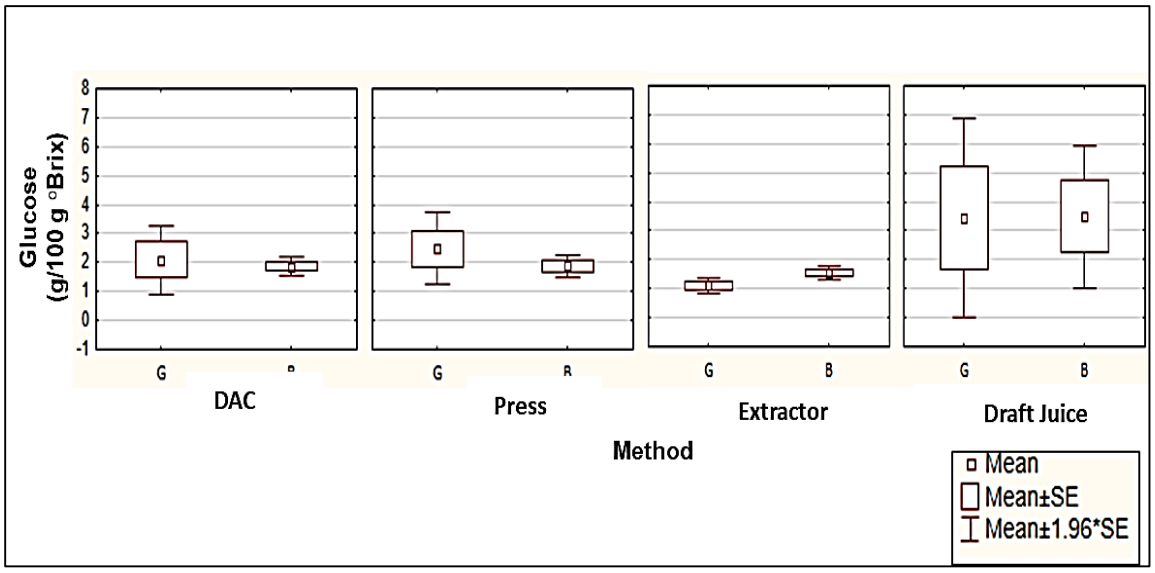

Figure 4-18: Box and whisker plot showing the comparison of cold digestion, press, and pilot extractor and draft juices for green and burnt sugarcane for glucose concentration. G - green sugarcane, B – burnt sugarcane, SE – standard error

For the fructose content, stronger correlations were observed for the press juice measurements with the draft juice measurements than for the DAC extract and pilot extractor

juice measurements (Figure 4-19). However, it must be noted that the concentrations of reducing sugars in all extracted juices were very low under the extraction conditions tested (Figure 4-19 and Figure 4-20).

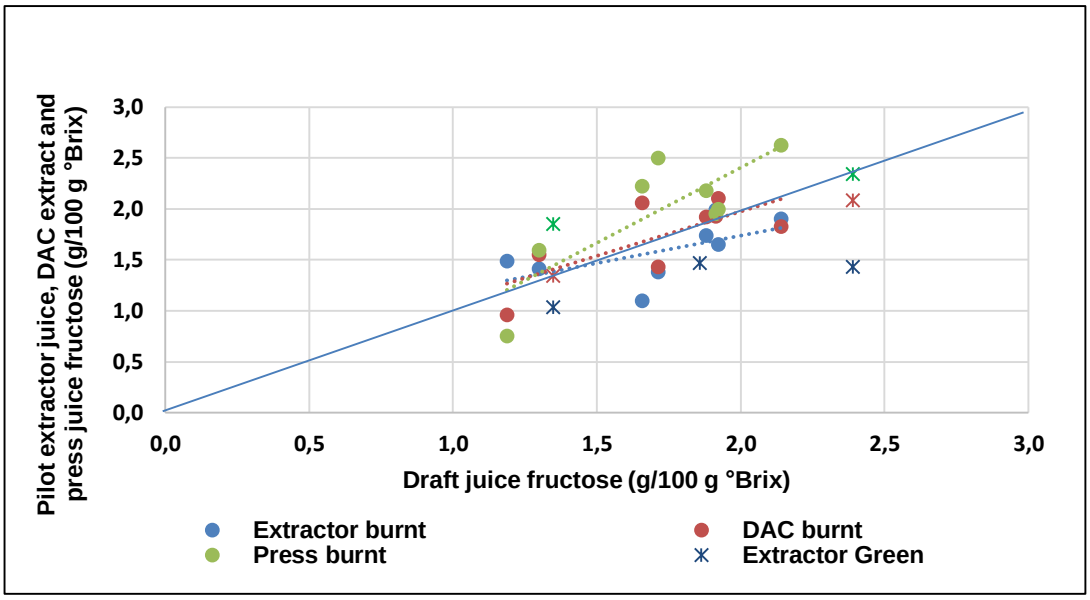

Figure 4-19:Fructose correlations for extracted juices against draft juice with the equivalence line represented by a solid blue line

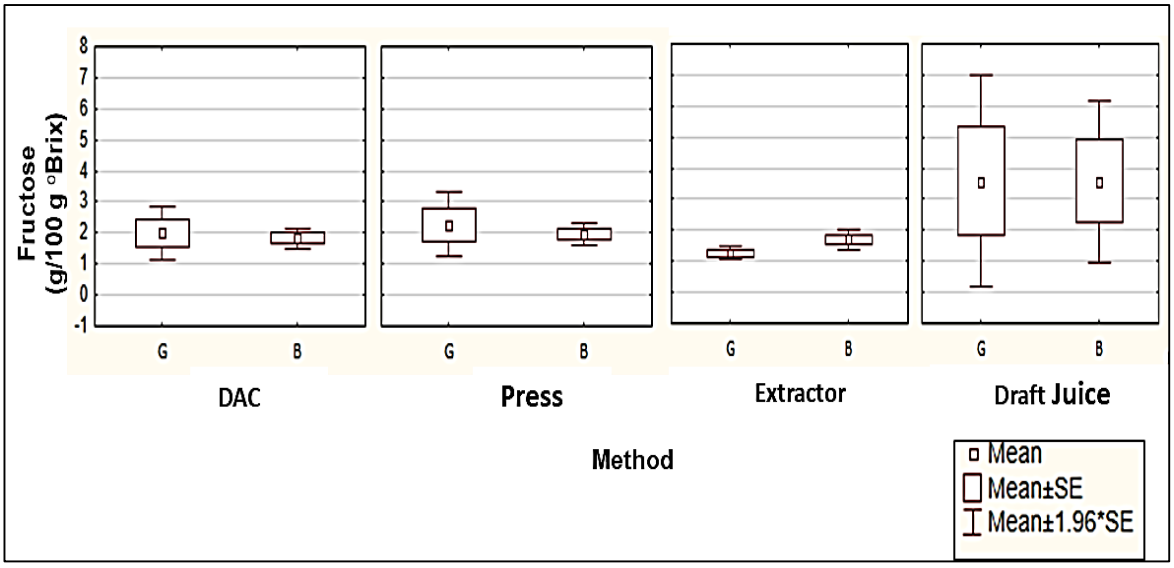

Figure 4-20:Box and whisker plot showing comparison of cold digestion, press, pilot extractor and draft juices for green and burnt sugarcane for fructose concentration. G - green sugarcane, B – burnt sugarcane, SE – standard error

The reducing sugar results showed some extreme outliers initially. These outliers were removed from the scatterplots presented in this section since, after careful investigation of the results, a decrease in the sucrose purity was observed for the samples circled in red and the data points corresponded to samples taken when there were noted stoppages in the diffuser. The scatterplots with the outliers are shown in Appendix B (and). This would have resulted in sucrose remaining in the diffuser for some time and thus decomposing into the reducing sugars.

The trend lines for the ash content in juice from the cold digester and press methods were closer to the equivalence line than the trend line for the ash content in juice from the pilot juice extractor (Figure 4-21). The pilot extractor juice measurements showed the highest ash content from all methods including the draft juice (Figure B-2 and Figure B-3). It was hypothesised that the conductivity ash in the DAC extract would be much higher than that in the juice of the pilot extractor and that the conductivity ash in the juice from the press would be lower than that in the juice from the pilot extractor (Barker and Davis, 2013).

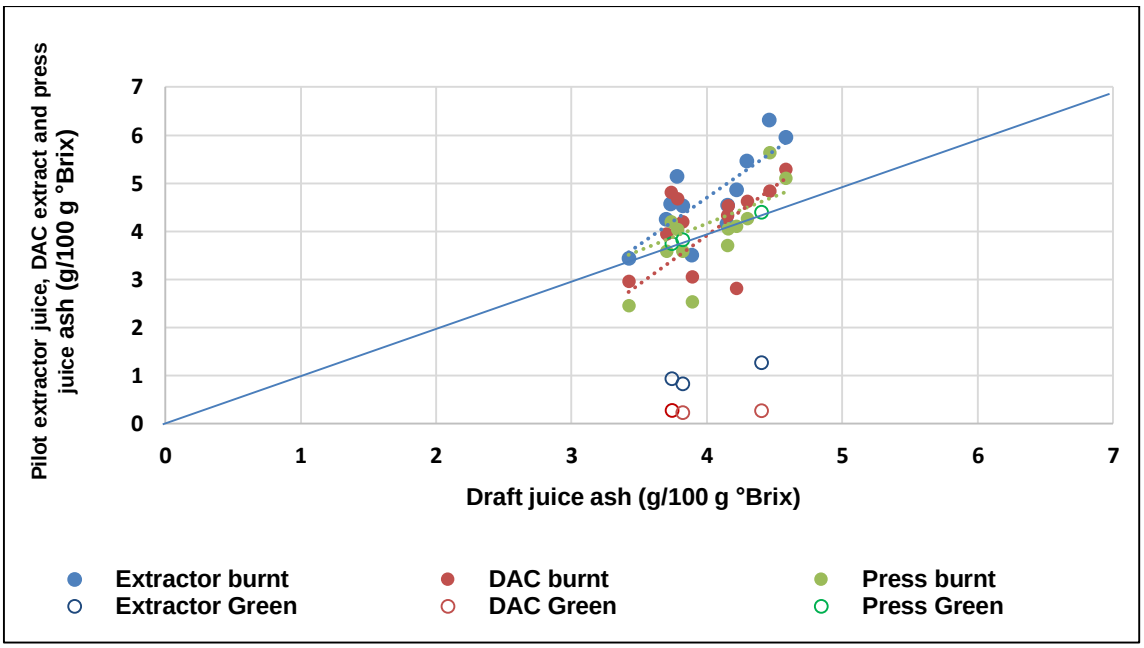

Figure 4-21:Conductivity ash correlations for extracted juices against draft juice with the equivalence line represented by a solid blue line

However, this was not the case and the reason for this anomaly requires further investigation. The method used for the conductivity ash analyses may have contributed to this anomaly as

the conductivity ash empirical equation was developed for a mixed juice of a higher brix (12-13%) than that obtained for the pilot extractor juice (2-3%). A further hypothesis is that the conductivity ash analytical method used for mixed juice analysis may not be the most suitable method of measuring ash in the pilot extractor juice due to the large dilution involved. The conductivity ash method requires a 20× dilution for mixed juice which is equivalent to about a 10× dilution for press juice, 40× dilution for DAC extracts and an 80×dilution for pilot extractor juice based on the respective brix concentrations. No significant differences between green sugarcane and burnt sugarcane values were noted for any of the extraction methods (Figure 4-22).

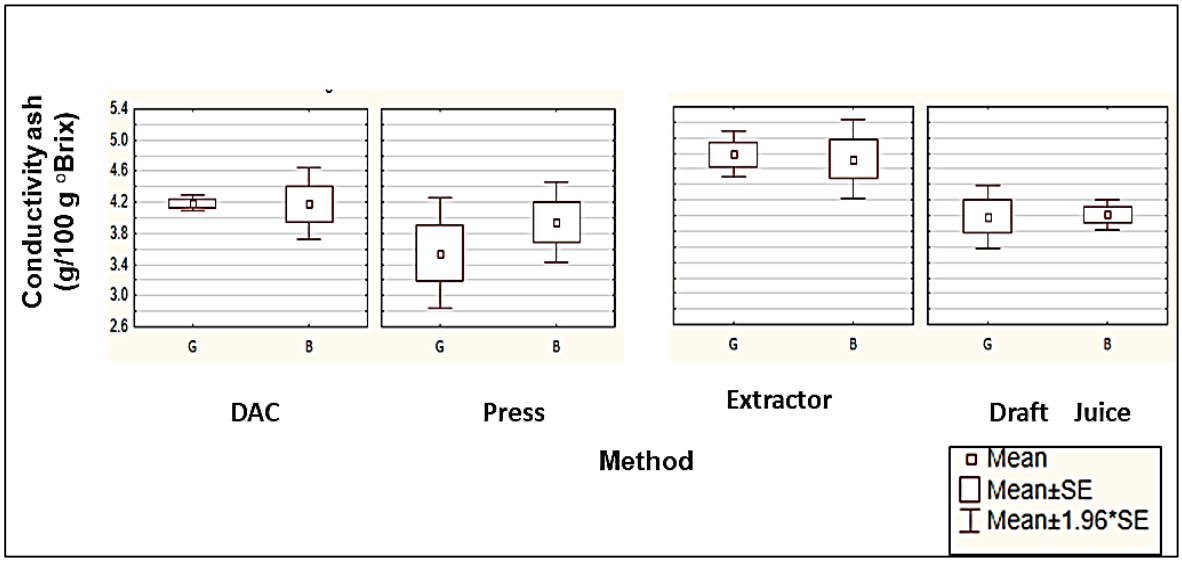

Figure 4-22:Box and whisker plot showing the comparison of cold digestion, press, pilot extractor and draft juices for green and burnt sugarcane for conductivity ash concentration. G - green sugarcane, B – burnt sugarcane, SE – standard error

The trend line representing the non-sucrose concentration of juice from the pilot juice extractor was much closer to the equivalence line compared to the trend lines obtained for the other two extraction methods. The pilot extractor also showed the strongest correlation with draft juice in this case (Figure 4-23). Larger variations in results were observed for green sugarcane for all extraction methods when compared to burnt sugarcane for the draft juice and press juices compared to the extractor juice and DAC extract (Figure 4-24).

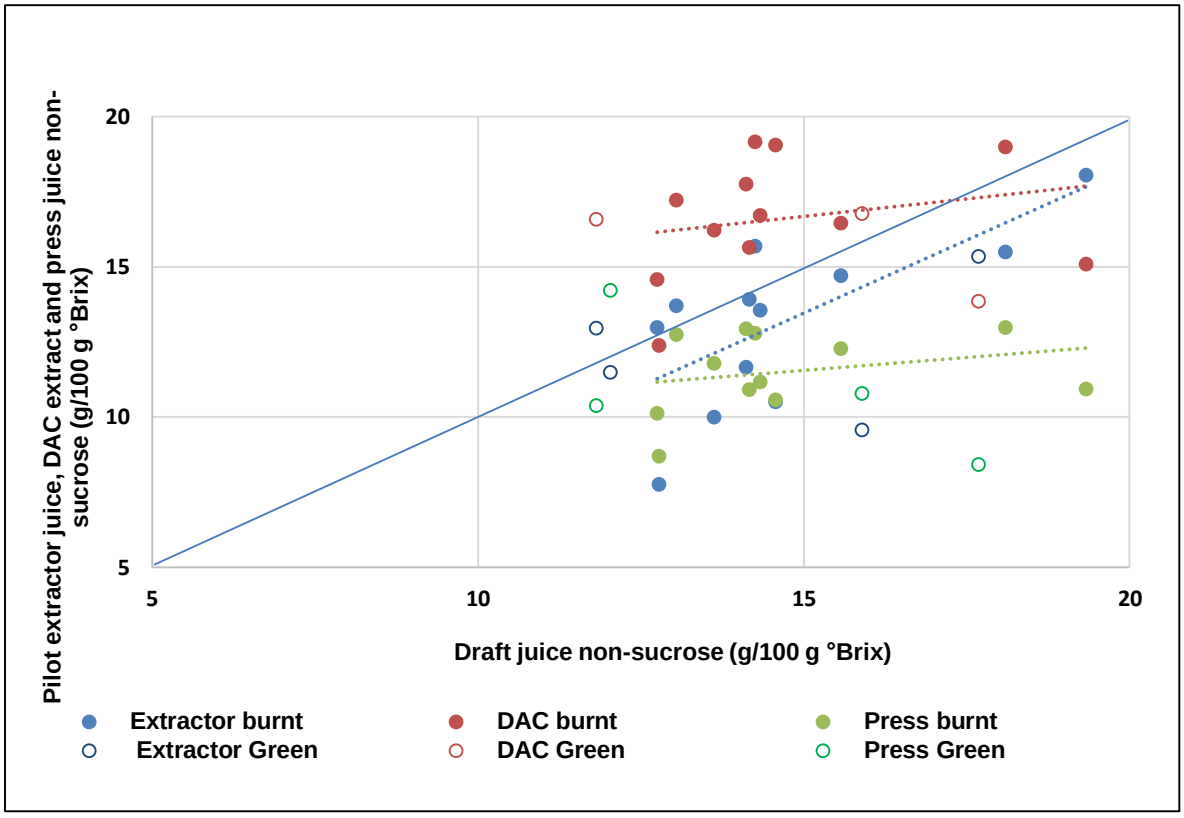

Figure 4-23:Non-sucrose correlations for extracted juices against draft juice with the equivalence line represented by a solid blue line

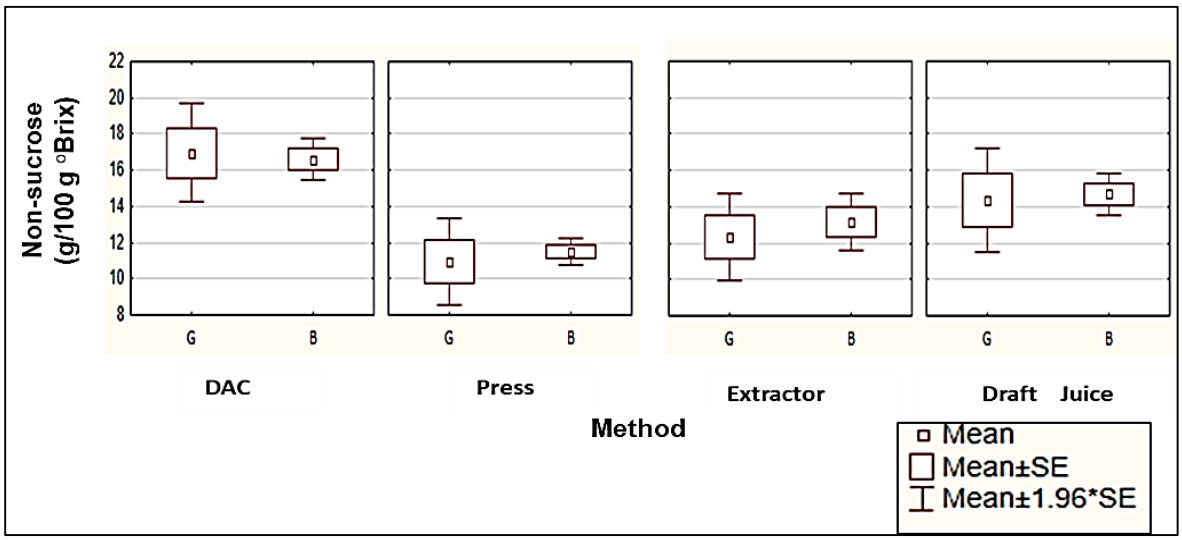

Figure 4-24:Box and whisker plot showing the comparison of cold digestion, press, pilot extractor and draft juices for green and burnt sugarcane for non-sucrose concentration. G - green sugarcane, B – burnt sugarcane, SE – standard error

Table 4-2 presents the Pearson correlation coefficients for all analyte concentrations between the three extraction methods with that of the draft juice for burnt sugarcane only. The correlations of the pilot extractor juice with draft juice were the strongest of all three methods for all of the analytes except for fructose concentration and conductivity ash. Although the analysis of juice from the pilot extractor correlated best of all three extraction methods tested with draft juice, the correlations overall were not considered strong. The moderate correlation coefficients may be attributed to the draft juice variations, which are a result of the counter-current juice flow and a large degree of mixing and recycling in the diffuser, which could not be controlled.

Table 4-2: **Correlation coefficients (for burnt sugarcane only) for different analytes and extraction methods compared to draft juice. Numbers in green indicate the best correlation while red shows the worst correlation of the three methods per analyte**

Analyte	Pilot extractor juice	DAC extract	Press juice
Colour	**0.42**	**0.20**	0.32
Gravity purity	**0.50**	**0.06**	0.07
Glucose/brix	**0.43**	0.30	0.26
Fructose/brix	**0.36**	0.54	**0.66**
Conductivity ash/brix	0.59	**0.23**	**0.61**
Non-sucrose/brix	**0.50**	**0.06**	0.07

There are many uncontrolled variables during diffuser operation that can influence the composition of the draft juice resulting in weaker correlations; therefore, it cannot be expected that any laboratory or pilot extraction method could exactly replicate the draft juice since these methods are not subjected to the same random variations. Any sample of draft juice is influenced by variations in conditions in the diffuser, during the period before sampling up to the longer periods of the sugarcane and juice mean residence times. By including sugarcane from only large consignments and sampling draft juice only when the last sugarcane from the consignment entered the diffuser, the juice sampled will be least influenced by other sugarcane consignments. Nevertheless, the conditions (temperature and

sugarcane bed saturation) will also have varied during the sugarcane consignment residence time in the diffuser, resulting in random sources of variance in juice composition. For this study, the diffuser draft juice is the reference material for the sugarcane consignments tested. In a diffuser, shredded sugarcane is repeatedly washed, but does not remain submerged for the entire extraction period, whereas the pilot extractor was designed to ensure complete submersion of the prepared sugarcane for the entire extraction period and allowed for more controlled experiments.

The GH diffuser, from which draft juice was sampled in this study, was set up to process burned sugarcane with low brown leaf content. The diffuser was not configured to handle green sugarcane and settings such as bed height and chain speed may have not been suitable for processing green sugarcane. Preparation equipment settings and diffuser imbibition flows and recycles have been selected to give good extraction for the expected shredded sugarcane consistency. The presence of significant quantities of brown leaf influences the performance of the preparation equipment and the density of the shredded sugarcane in the diffuser. Shredder and diffuser settings are not adjusted for every sugarcane consignment, and therefore, when a large change in sugarcane preparation and bed density occurs, diffuser extraction performance is also expected to change. Therefore, in addition to random variations during normal, relatively steady operation of the diffuser, the variance in draft juice quality when processing green sugarcane with brown leaf is expected to be even higher which was observed in the study. If extraction is negatively influenced under these conditions, the draft juice may be expected to have lower concentrations of the hard-to-extract constituents such as colour, ash and reducing sugars than would be obtained if the sugarcane preparation equipment and diffuser were set up specifically to deal with green sugarcane.

For the reasons mentioned above, it was decided that the objectives of the validation phase,viz., to validate the pilot juice extractor against a full-scale diffuser and to compare the resultant juices with the juices extracted from the two established methods against the draft juice from a diffuser, was achieved. The moderate correlations for all methods plotted against the draft juice were accepted and considered sufficient to move to the next phase of the study. It is believed that stronger correlations are not achievable, given the circumstances

described above and performing further work would have not assisted in establishing additional conclusions as to what had already been established in the study.

4.3 Brown leaf experiments

The final stage of the project objectives was to:

• Investigate the effects of variety, type (green or burnt sugarcane) and varying quantities of brown leaf addition on the quality of juice extracted from a pilot juice extractor for four different sugarcane varieties

• Investigate the effects of variety, type (green or burnt sugarcane) and varying quantities of brown leaf on the percolation rate for four different sugarcane varieties

The results from the aforementioned objectives will be used to understand and estimate the impact of co-processing green sugarcane with brown leaf in a commercial diffuser. The data obtained from the experimental trials were analysed using TIBCO Statistica ™ (version 13.4.0.14) software. The full factorial experimental design comprising of 48 tests is shown in Appendix C: Table C-3. Sugarcane variety was varied at four levels (four different varieties) whereas brown leaf addition for green sugarcane was tested at three levels (low, medium and high; 0, 7.5 and 15%). These levels were determined by taking the average of the 4 amounts of brown leaf content determined per variety (15%) as shown in Appendix C: Table C-1 and using that as a maximum level whereas the middle level was taken as half that amount (7.5%). Burnt sugarcane was also tested for comparisons with green sugarcane. The green sugarcane at the lowest level of brown leaf (0%) was used as the control in the experiments. Cold digestion tests were performed to determine the maximum extraction that was achievable per a variety and condition giving a total of 16 cold digestion tests. In addition, a control test was performed per a variety to track sugarcane deterioration from the time of harvest, collection and preparing the sugarcane to before the actual experiments were conducted. The details of the sugarcane varieties used, and their respective purity deterioration checks are shown in Appendix C: Table C-2.

All burnt sugarcane samples were noted to display occurrences of fungal growth which was expected since burning is believed to destroy the protective waxy sugarcane rind (cracking of rind) thereby exposing the internals of the stalk to moisture. In addition, the N39 variety showed the presence of eldana infection (pest infection which damages the stalk and leads to loss biomass and sucrose content) and some of the N47 stalks were noted to be quite thick. The N16 stalks were stored in a fridge after cleaning to preserve them since the experimental tests ran over a few days. The highest purity drop was observed for variety N39 and was expected due to the eldana infection, followed by N16 which was also expected due to it being stored for the longest number of days prior to usage. The N47 purity gain can be explained by the fact that the stalks varied quite differently, which would have led to possible variation in sample size or that the sugarcane was mature (growth stops, and sucrose concentration increases).

The raw data results for the brown leaf trials (Pilot juice extractor, sugarcane density & percolation tests) and the DAC extract result, as well as the control tests, are presented in Appendix C: Table C-5 and Table C-6 respectively. The sample calculations for the sugarcane density and percolation results are also shown in Appendix C. Box and whisker plots were plotted to graphically represent the data and the standard deviations. The statistical tests were conducted for green and burnt sugarcane across all sugarcane varieties to meet the first objective and thereafter for green sugarcane only with the various levels of brown leaf addition for the different varieties to meet the second objective.

ANOVA statistical analyses was performed at a 95% confidence interval to determine whether the varietal groups tested for green cane with no brown leaf were statistically different for each of the analytes. It also determined if statistically significant differences were present between green and burnt sugarcane with no brown leaf addition and for the different levels of brown leaf addition for green sugarcane only. However, the ANOVA tests did not indicate which of the groups tested were different. An LSD (least significant difference) test was performed to compare the means between each group. Regression graphs were plotted, thereafter at a 95% confidence interval to establish the linear fit correlations and their respective equations.

4.3.1 Varietal effects

The analysis of variance (ANOVA) test was performed at a 95% confidence interval to initially determine if sugarcane varieties (green with no brown leaf added) were statistically different from each other by comparing the concentrations of each of the analytes present in the extracted juice, the sugarcane densities and the percolation rates.

The ANOVA test revealed that for some variables the varieties are statistically different from each other for all the analytes (P- values below 0.05) marked in red in Table 4-3.

Table 4-3: ANOVA test indicating whether green sugarcane varieties (with no brown leaf) had a significant effect on the quality of extracted juice, sugarcane density and percolation rate (P-values < 0.05)

Dependent variable	P-value
Gravity Purity	0.0294
Fructose/brix	0.0877
Glucose/brix	0.2189
Conductivity ash/ brix	0.0157
Colour	0.0001
Sugarcane density	0.1430
Percolation rate	0.0140

4.3.1.1 Gravity purity

Significant differences between varieties were observed in terms of gravity purity. This was expected after inspecting the sugarcane post-harvest. As mentioned earlier, variety N39 had very thin stalks with evidence of eldana infestation while N47 had very thick stalks. The box and whisker plots for gravity purity for green sugarcane without brown leaf are shown in Figure 4-25. The results confirm that N39 has the lowest purity as expected. The large variability of N47 can be attributed to the noticeable differences in stalk thickness.

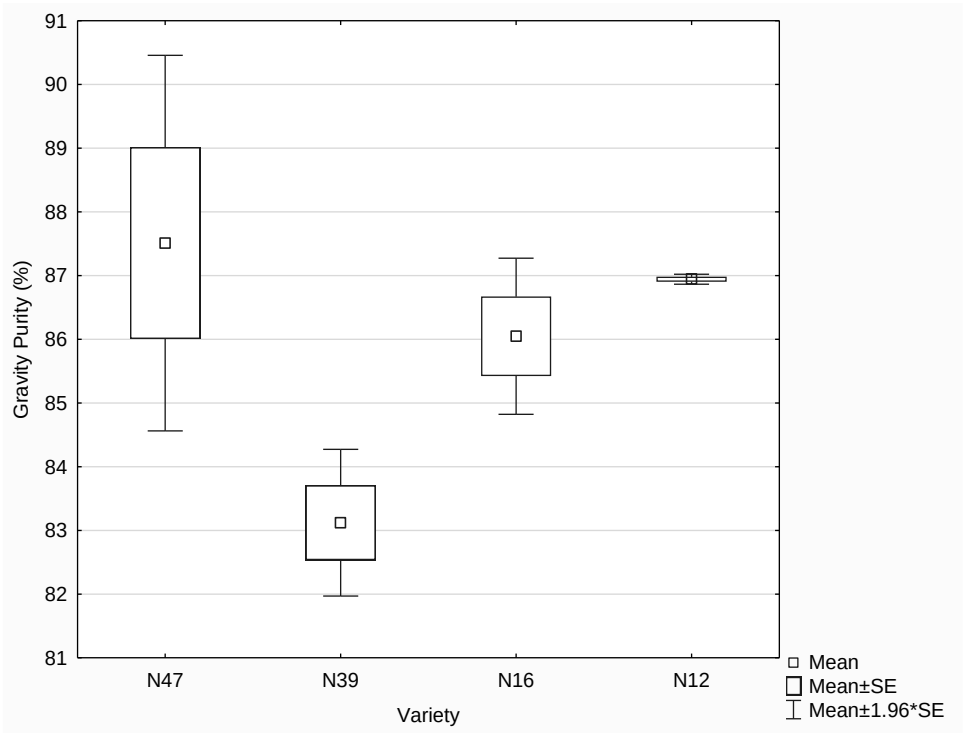

Figure 4-25: Gravity purity of the different varieties (green with no brown leaf added) that were used in the investigations

4.3.1.2 Colour

Since the sugarcane varieties were chosen for their different colour characteristics, it was expected that the colour difference between varieties would be significant. Figure 4-26 shows the difference between varieties for green sugarcane with no brown leaf. Variety N47, selected as the low colour variety and N12 selected as the high colour variety were the highest and lowest colour varieties, respectively, as predicted, while N12 and N16 selected as medium colour varieties, had colour values that ranged between those of variety N47 and variety N12.

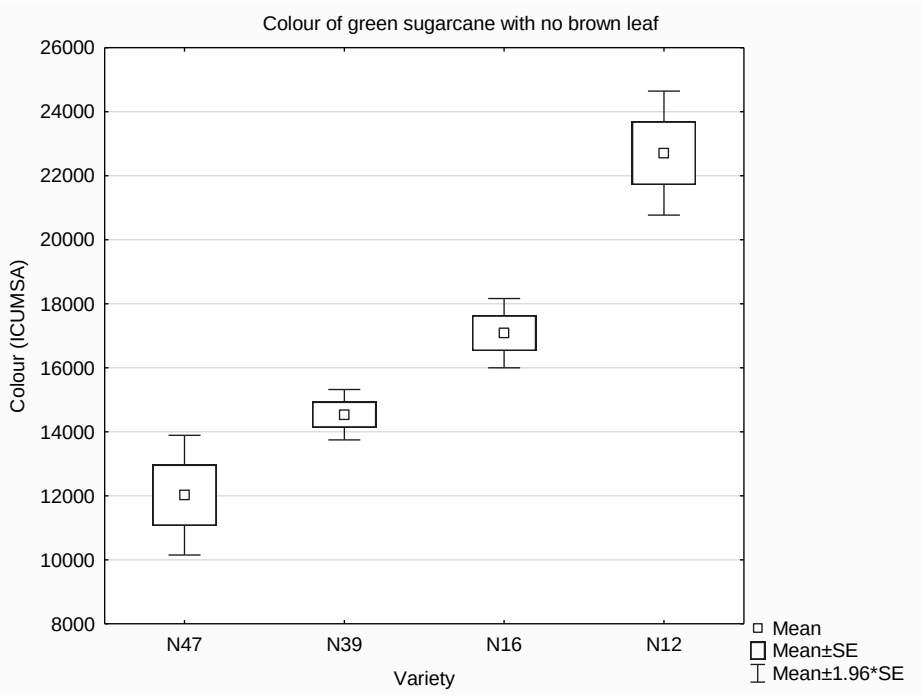

Figure 4-26: ICUMSA Colour of the different varieties (green with no brown leaf added), that were used in the investigation

4.3.1.3 Conductivity ash

Significant differences were found for conductivity ash (Figure 4-27) which can be expected since the variety can have a significant effect on ash even from the same geographical location (Barker and Davis, 2005).

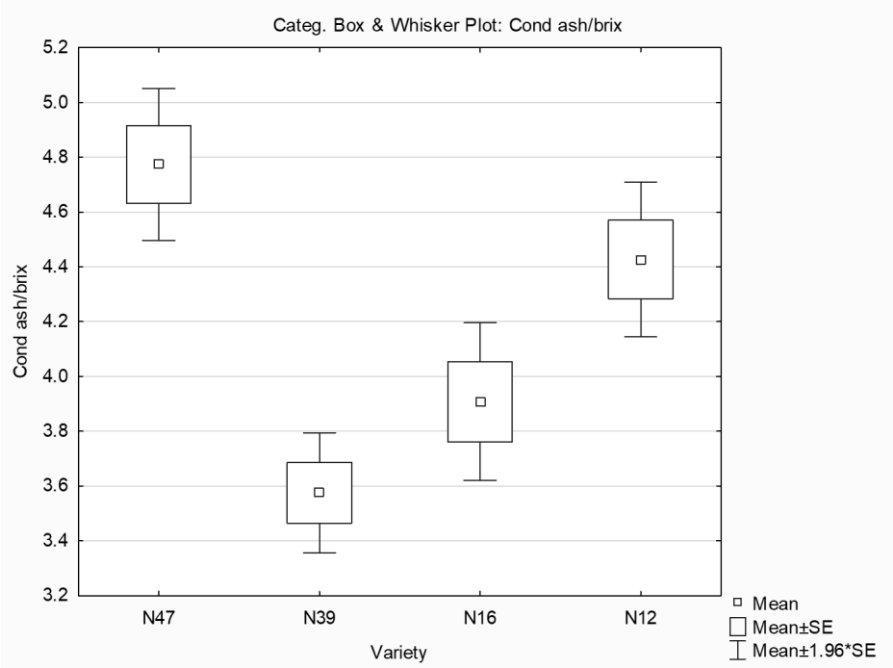

Figure 4-27: Conductivity ash of the different varieties (green with no brown leaf added), that were used in the investigations

4.3.1.4 Percolation rate and density

Significant differences were also noted between varieties for percolation rate (Figure 4-28), as previously reported. This is not unexpected as previous work has shown that variety can have a significant effect on preparation and percolation behaviour (Loubser and Barker, 2011). However, there were unexpectedly no significant differences in sugarcane density, which is an indirect measure of percolation behaviour and hence the box and whisker plot is omitted.

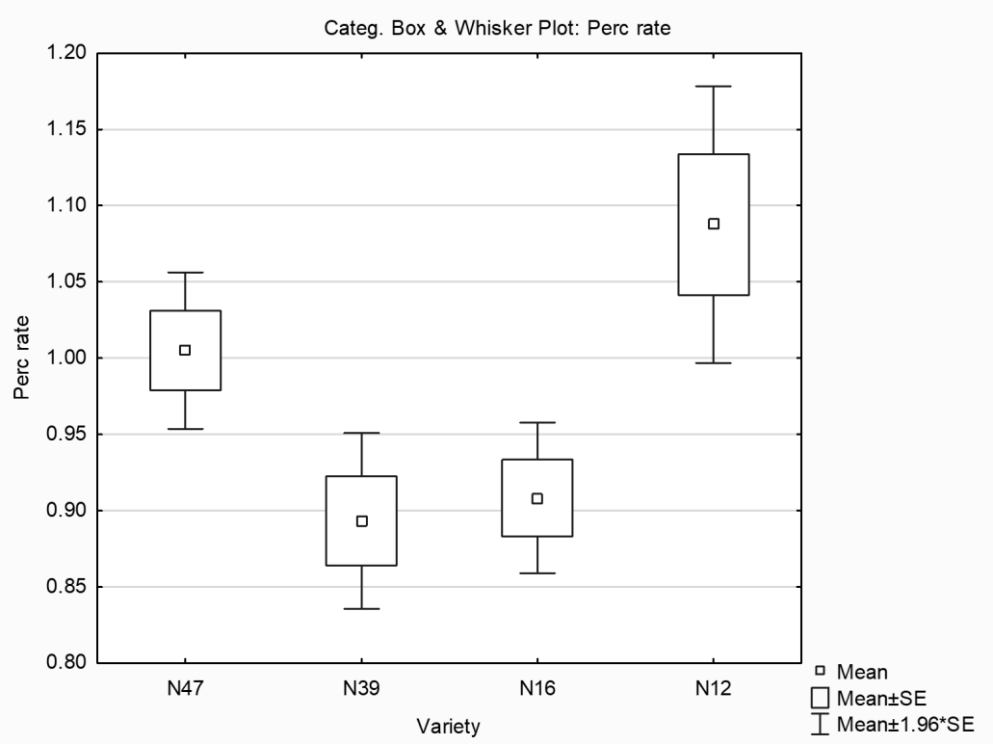

Figure 4-28: Percolation rate of the different varieties (green with no brown leaf added), that were used in the investigations

4.3.1.5 Reducing sugars

No significant differences between varieties were found for glucose and fructose and hence the box and whisker plots are omitted. This may be due to large analytical uncertainties associated with analysis of reducing sugars at the low concentrations of extractor juices.

4.3.2 Green versus burnt sugarcane (no brown leaf)

An ANOVA test was also performed at a 95% confidence interval to determine if burnt and green sugarcane with no brown leaf are statistically different from each other in terms of the concentrations of the analytes present in the extracted juice as well as on the sugarcane density and percolation rate. The ANOVA test indicated that burnt and green sugarcane were statistically different from each other for all the analytes (P- values below 0.05) marked in red in Table 4-4. Burnt and green sugarcane were identified as being statistically significant for only gravity purity, colour and non-sucrose on brix.

Table 4-4: ANOVA test showing statistical effects of burnt and green sugarcane on the quality of juice extracted, sugarcane density and percolation rate (P values < 0.05)

Variable	P-value
Gravity purity %	0.004019
Colour	0.010685
Fructose/brix	0.058380
Glucose/brix	0.820720
Conductivity ash/brix	0.388817
Reducing sugars/ash	0.830646
Non-sucrose/brix	0.004019
Sugarcane density	0.333912
Percolation rate	0.212821

An LSD test was performed to compare the specific sugarcane varieties per response and determine which variety differed statistically for burnt and green sugarcane. The results are summarised in Table 4-5 where a statistical significance of the means for a confidence interval of 95% ($p < 0.05$) was determined. The LSD tests indicated specifically which varieties showed no differences between burnt and green sugarcane for the specific analytes. The results are discussed collectively with the box and whisker plots (Figure 4-29 to Figure 4-37)

Table 4-5: LSD test showing statistical differences of means for burnt and green sugarcane on the analytes across all sugarcane variety groups tested (P values < 0.05)

Analytes	Sugarcane Variety			
	N12	N39	N47	N16
Gravity purity %	✓	✓	✓	X
Colour	X	✓	✓	✓
Fructose/brix	X	X	✓	X
Glucose/brix	X	X	X	X
Conductivity ash/brix	✓	X	X	X
Reducing sugars/ash	X	X	X	X
Non-sucrose/brix	✓	✓	✓	X
Sugarcane density	X	X	X	X
Percolation rate	X	X	X	X

Key:

✓ = statistically significant

X = not statistically significant

4.3.2.1 Gravity purity

For gravity purity (Figure 4-29) the burnt and green sugarcane was statistically significant for all varieties except N16, according to the LSD tests (Table 4-5). Green sugarcane showed higher purities than burnt sugarcane overall. It is expected that the green sugarcane should show higher purities than burnt sugarcane since burning is believed to crack the protective waxy layer of the sugarcane and thus exposes the internals of the sugarcane to the moisture in air, resulting in deterioration and thereby reducing the purity of the sugarcane. The N16 green sugarcane may have experienced higher levels of deterioration due to being stored for a longer time than the other varieties before being tested. This would have caused a drop in the purity of the green sugarcane making it very close to that of the purity in burnt cane, thereby reducing the difference in the mean values between burnt and green sugarcane. The lower purities for both burnt and green sugarcane for N39 as compared to all other varieties may be a result of deterioration due to the eldana infestation.

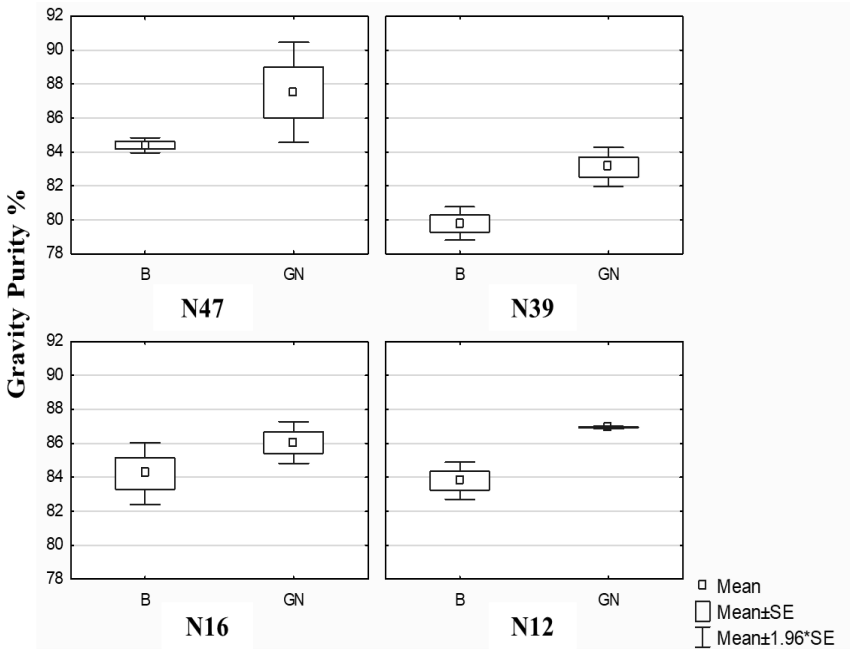

Figure 4-29: Box and whisker plot showing the comparison of gravity purity % for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

4.3.2.2 Colour

For colour, the burnt and green sugarcane was statistically different from each other for all varieties except N12, according to the LSD tests (Table 4-5). Burnt sugarcane showed higher colour content than green sugarcane overall (Figure 4-30). It is hypothesised that the burning of sugarcane adds to colour (Reid and Lionnet, 1989). N12 is known to be a high colour variety and hence the colour added to the burnt sugarcane by the degree of burning may have not been as large as the concentrations of colour that is already present in the green sugarcane when compared to the other varieties.

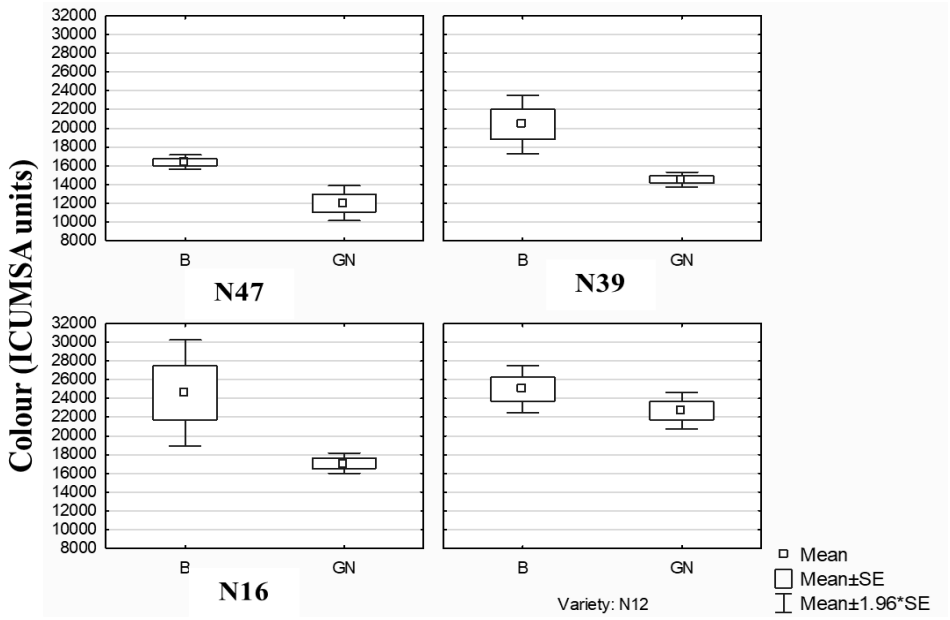

Figure 4-30: Box and whisker plot showing the comparison of colour for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For fructose content, the burnt and green sugarcane were not statistically significant across all varieties, except for N47 (Table 4-5). Careful examination of the box and whisker plot (Figure 4-31) reveals that a large overlap exists for the results and the means are very similar. For this reason, a definite result may not be concluded as to whether burnt or green sugarcane shows higher fructose concentrations. It is assumed that the low concentrations of this component may be subject to poor analytical precision. The different result observed for N47 may be attributed to the variation of the green sugarcane stalks in the sample, which could have caused it to appear as significantly different. Similarly, no differences were observed for all varieties for burnt and green sugarcane for glucose content (Figure 4-32) for the same reasons.

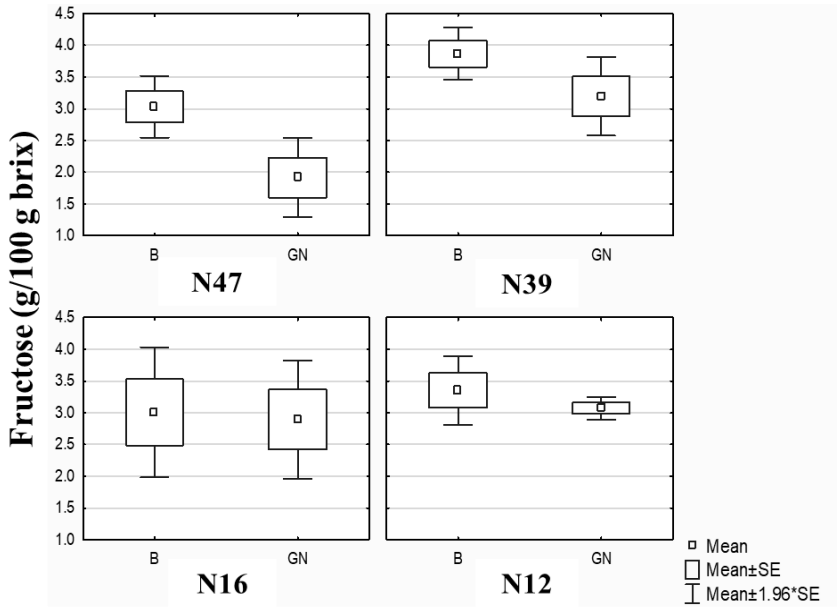

Figure 4-31: Box and whisker plot showing the comparison of fructose content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

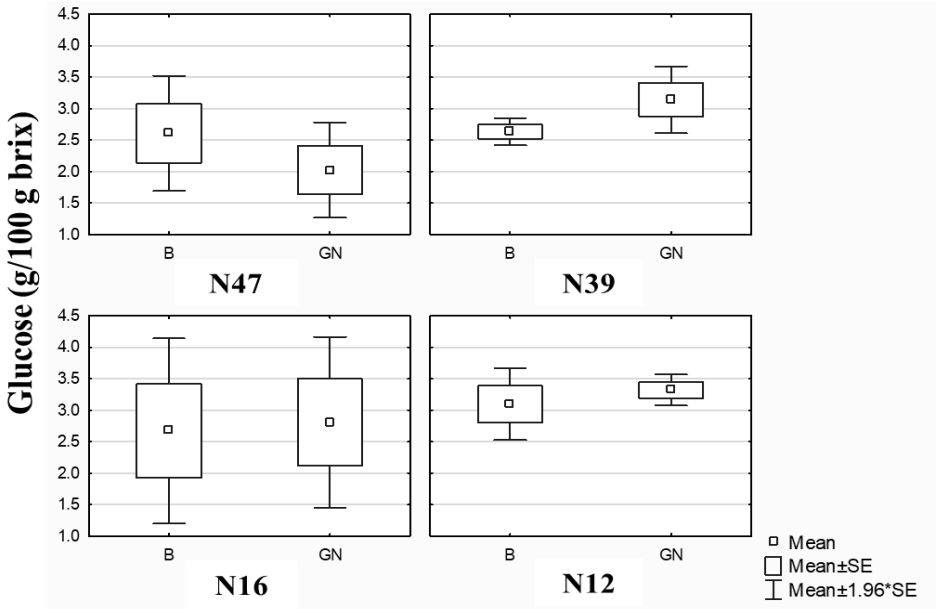

Figure 4-32: Box and whisker plot showing the comparison of glucose content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For conductivity ash, the burnt and green sugarcane were not statistically different from each other for all varieties except N12 (Table 4-5).No reliable trend was observed in this case (Figure 4-33). Juices extracted from burnt sugarcane are believed to show higher levels of ash (Reid and Lionnet 1989). Conductivity ash levels are also believed to increase with sugarcane deterioration, and it is possible that the deterioration of the green sugarcane for N39, N47 and N16 caused ash levels to increase such that the concentrations were very similar to that measured in burnt sugarcane. Alternatively, the method used for conductivity ash may have not been the ideal representation of the ash levels present in the juice, as was experienced in the validation phase.

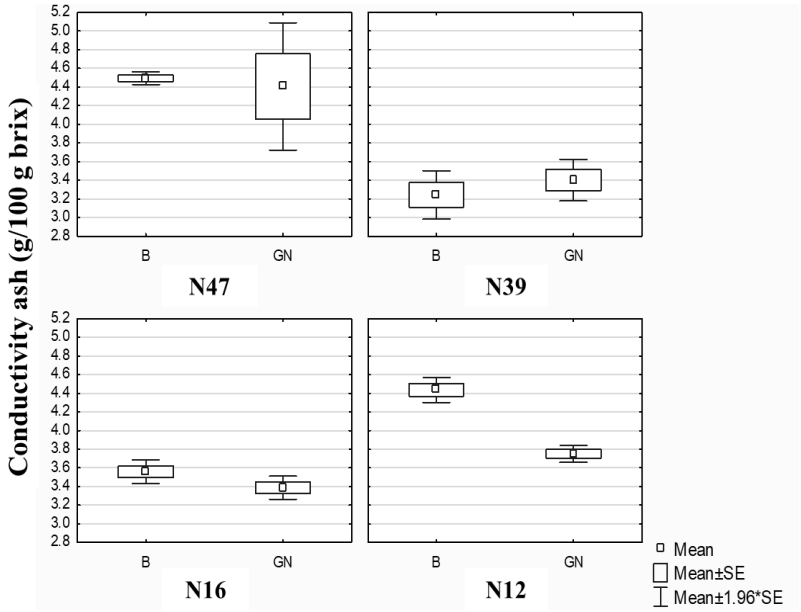

Figure 4-33: Box and whisker plot showing the comparison of conductivity ash content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

Reducing sugar: ash ratios (Figure 4-34) are largely dependent on the validity of the reducing sugar and ash results and since those results were not conclusive, the result of not finding any statistically significant differences, in this instance, was accepted.

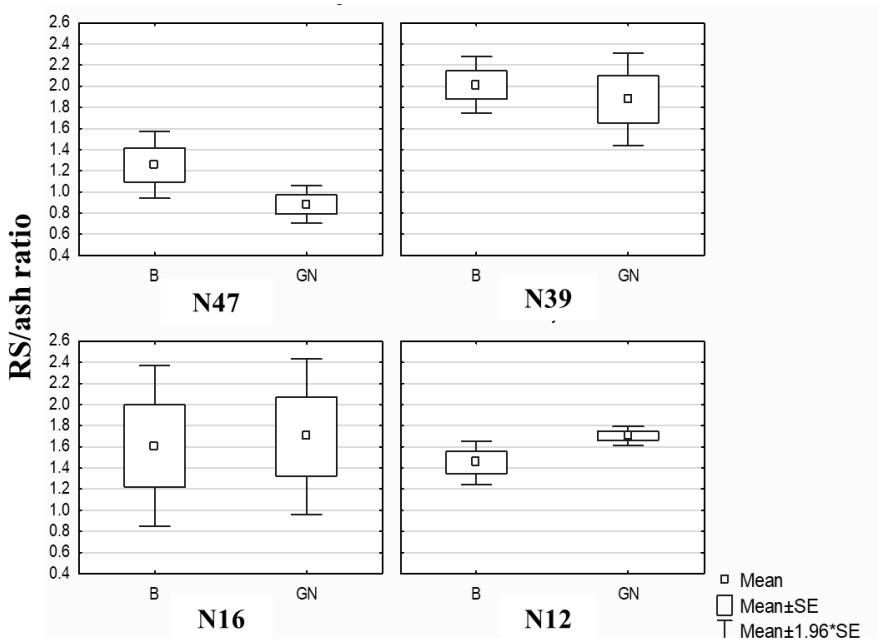

Figure 4-34: Box and whisker plot showing the comparison of reducing sugar: ash ratio for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For non-sucrose content, the same result as observed with gravity purity was observed where all varieties showed statistical differences between burnt and green sugarcane, except N16 (Table 4-5). Overall, burnt sugarcane showed higher levels of non-sucrose than green sugarcane (Figure 4-35). Non-sucrose content represents impurities. A decrease in purity would infer that there is an increase in impurities. Since the green sugarcane for N16 was previously assumed to have showed lower purities, making it similar to the burnt sugarcane, it should be expected that the impurities would be similar for the burnt and green sugarcane.

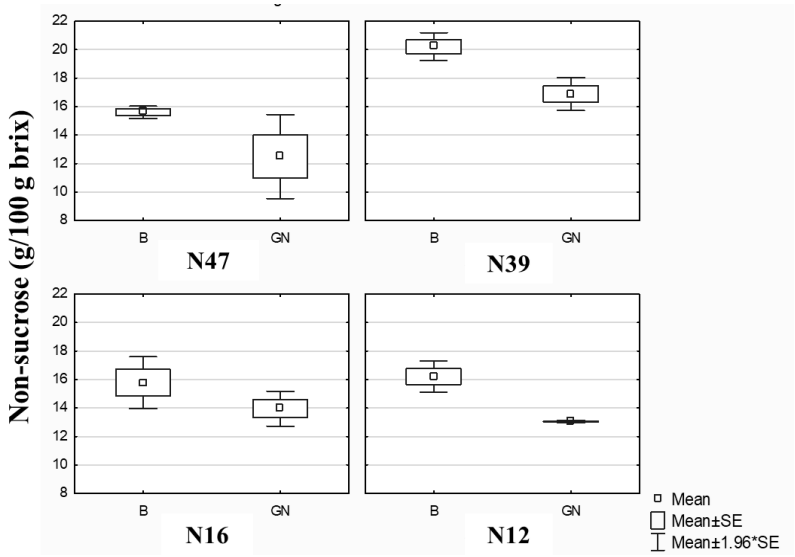

Figure 4-35: Box and whisker plot showing the comparison of non-sucrose content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For sugarcane density (Figure 4-36), it is expected that burnt sugarcane and green sugarcane without brown leaf would have the same density and the same percolation rates (Figure 4-37) as sugarcane density is expected to affect the percolation rates. The LSD tests (Table 4-5) was in agreement and showed that no statistically significant difference is noted for burnt and green sugarcane across all varieties for sugarcane density and percolation rates, hence, the results are acceptable.

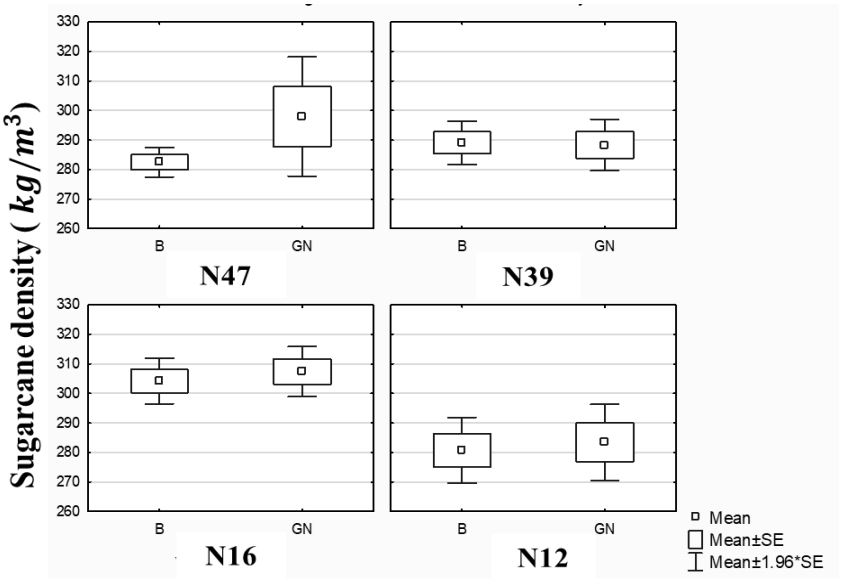

Figure 4-36: Box and whisker plot showing the comparison of sugarcane density for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

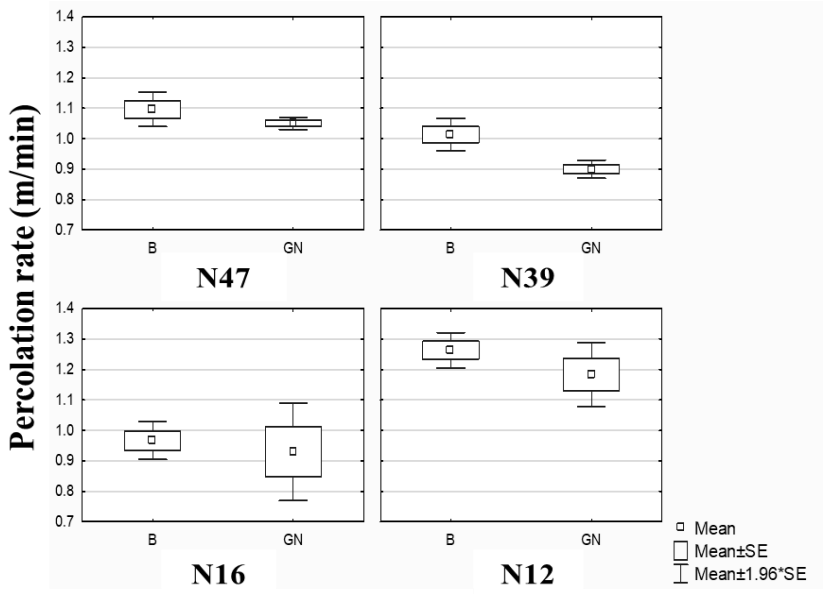

Figure 4-37: Box and whisker plot showing the comparison of percolation rate for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

4.3.3 Effect of different levels of brown leaf across different varieties

An ANOVA test was performed at a 95% confidence interval to determine if the different levels of brown leaf added, were statistically different from each other in terms of the concentrations of the analytes present in the extracted juice as well as on the sugarcane density and percolation rate.

The ANOVA test indicated that the different brown leaf levels are statistically different from each other for all the analytes (P- values below 0.05) marked in red in (Table 4-6). Only the reducing sugar content and reducing sugar: ash ratio was not identified as being statistically different for the different levels of brown leaf added.

Table 4-6

Table 4-6: ANOVA test showing statistical effects of brown leaf added to green sugarcane on the extracted juice quality, sugarcane density and percolation rate (P values < 0.05)

Variable	P-value
Gravity purity %	0.000112
Colour	0.000000
Fructose/brix	0.165614
Glucose/brix	0.231807
Conductivity ash/brix	0.000396
Reducing sugars/ash	0.804129
Non-sucrose/brix	0.000112
Sugarcane density	0.000000
Percolation rate	0.006874

An LSD test was then performed to compare the specific sugarcane varieties per analyte and determine which variety differed for the different levels of brown leaf added. The results are summarised in Table 4-7 where a statistical significance in means for a confidence interval of 95 % ($p < 0.05$) is shown. The LSD tests indicated specifically which varieties showed no differences for the different levels of brown leaf added for the specific analytes. Level interactions of none to medium (NM= 0 to 7.5%), none to high (NH= 0 to 15 %) and medium

to high (MH =7.5 to 15%) were analysed. All of the varieties tested displayed significant differences for colour and sugarcane density at all brown leaf -level interactions. The specific effects are discussed collectively with the box and whisker plots (Figure 4-38 to Figure 4-46).

Table 4-7: LSD test showing statistical differences of means for different levels of brown leaf added to green sugarcane on the analytes across all sugarcane variety groups tested (P values < 0.05)

Analytes	Sugarcane Variety											
	N12			N39			N47			N16		
Levels of brown leaf	NM	NH	MH	NM	NH	MH	NM	NH	MH	NM	NH	MH
Gravity purity %	✓	✓	✓	X	✓	X	X	✓	✓	✓	✓	✓
Colour	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Fructose/brix	X	X	X	✓	X	X	X	X	X	X	X	X
Glucose/brix	X	X	X	X	X	X	X	X	X	X	X	X
Conductivity ash/brix	✓	✓	X	X	✓	X	X	✓	✓	✓	✓	✓
Reducing sugars/ash	X	X	X	X	X	X	X	X	X	X	X	X
Non-sucrose/brix	✓	✓	✓	X	✓	X	X	✓	✓	✓	✓	✓
Sugarcane density	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Percolation rate	✓	✓	X	X	X	X	X	✓	X	X	X	X

Key:

✓ = statistically significant

X = not statistically significant

N- none (0 %), M -medium (7.5 %), H- high (15 %)

4.3.3.1 Gravity purity

For gravity purity, statistically significant differences were observed for all levels of brown leaf for both N12 and N16 varieties (Table 4-7). N39 showed significant differences for the none to high level only whilst N47 showed significant differences for the none to high and medium to high levels (Table 4-7). Increases in brown leaf content are expected to reduce the purities of extracted juices (Rein, 2007). This trend was observed overall for all varieties with the highest level of brown leaf corresponding to the lowest purity (Figure 4-38).

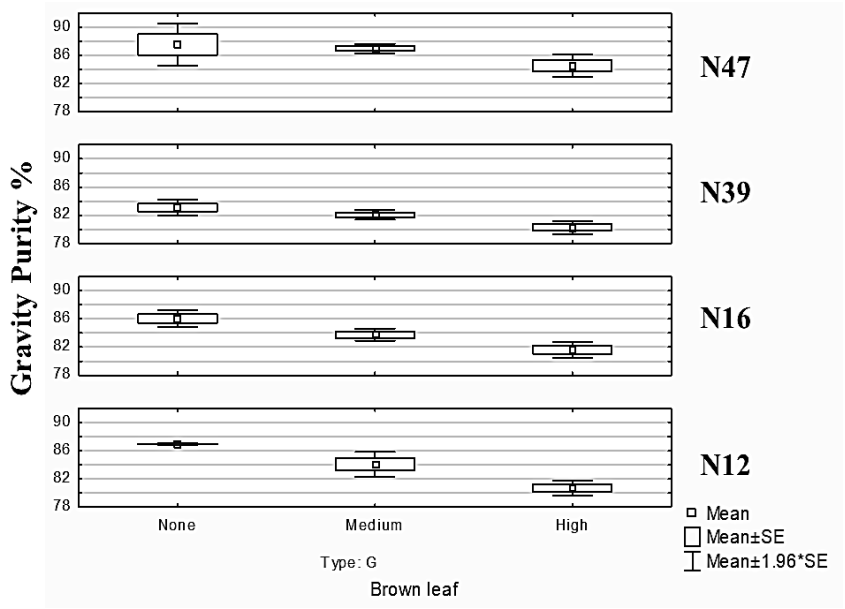

Figure 4-38: Box and whisker plot showing the comparison of gravity purity % for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

4.3.3.2 Colour

For colour, statistically significant differences were observed across all levels of brown leaf and all varieties (Table 4 -7). The addition of brown leaf (which is high in colour) is expected to add to colour in extracted juices (Lionnet, 1985). It is clear that an increase in brown leaf concentration increased the colour (Figure 4-39).

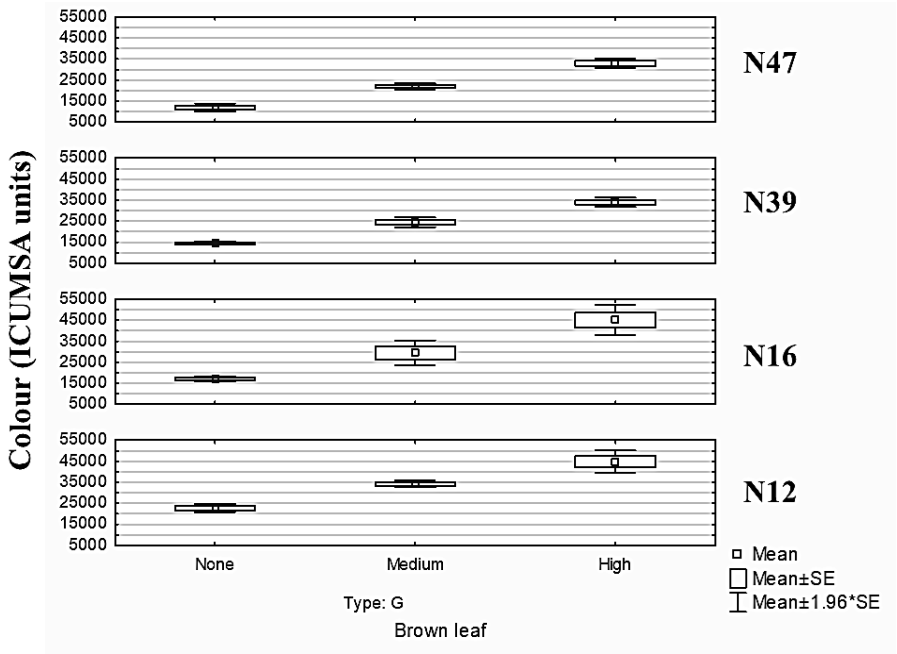

Figure 4-39: Box and whisker plot showing the comparison of colour for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For fructose content and glucose content (Table 4-7), no statistically significant differences were observed between all levels of brown leaf for all varieties. The individual effect of brown leaf content only on fructose and glucose concentrations in extracted juices are not known and have been reported in most studies as an effect of a combination of brown leaf and tops. The concentration of reducing sugars in brown leaf is considered to be close to zero as the leaves have died. It is also assumed that the concentrations of these components were rather small considering the dilute juices and that the analytical precision would be poor, hence, no definite conclusions can be drawn in this regard.

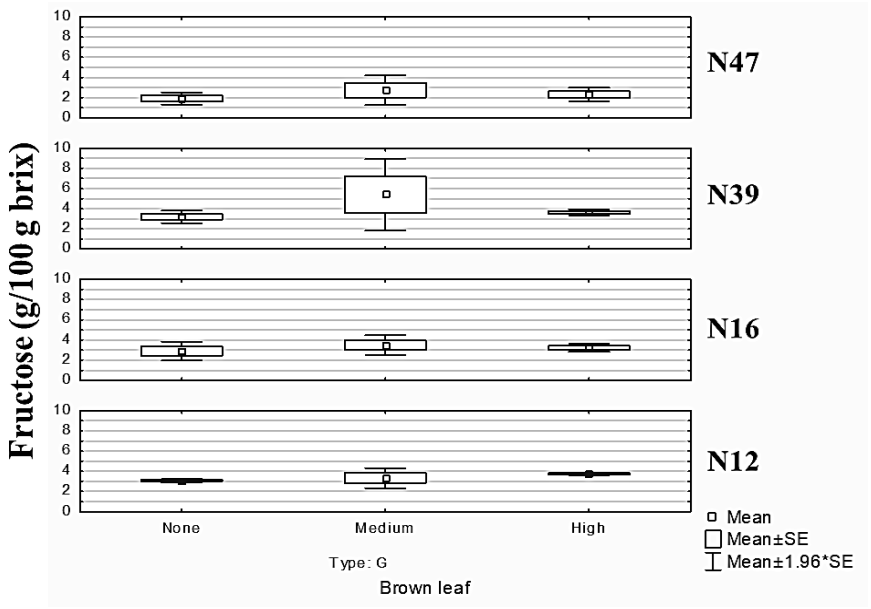

Figure 4-40: Box and whisker plot showing the comparison of fructose content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

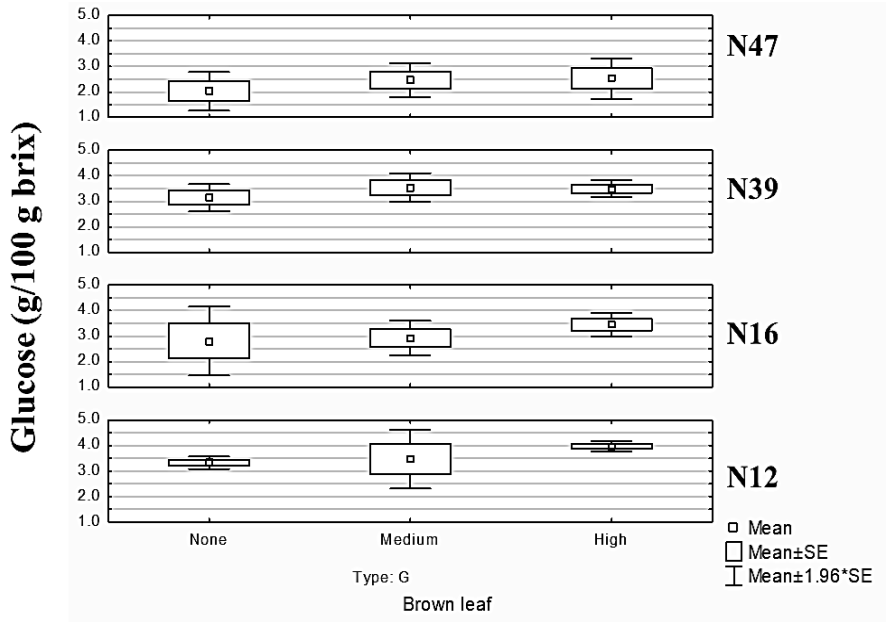

Figure 4-41: Box and whisker plot showing the comparison of glucose content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For conductivity ash, statistically significant differences were observed for most of the brown leaf levels across most of the varieties. There was a clear observation that an increase

in the brown leaf content, increased the conductivity ash content (Figure 4-42) and the result is an expected result since brown leaf is said to contribute to higher ash levels (Reid and Lionnet, 1989). N39 showed no statistical significance for none to medium and medium to high levels (Table 4-7). N39 showed higher levels of deterioration due to the eldana infection and it is expected that conductivity ash levels will increase with deterioration. This increase could have been to the extent that the conductivity ash increases caused by brown leaf, were not as significant.

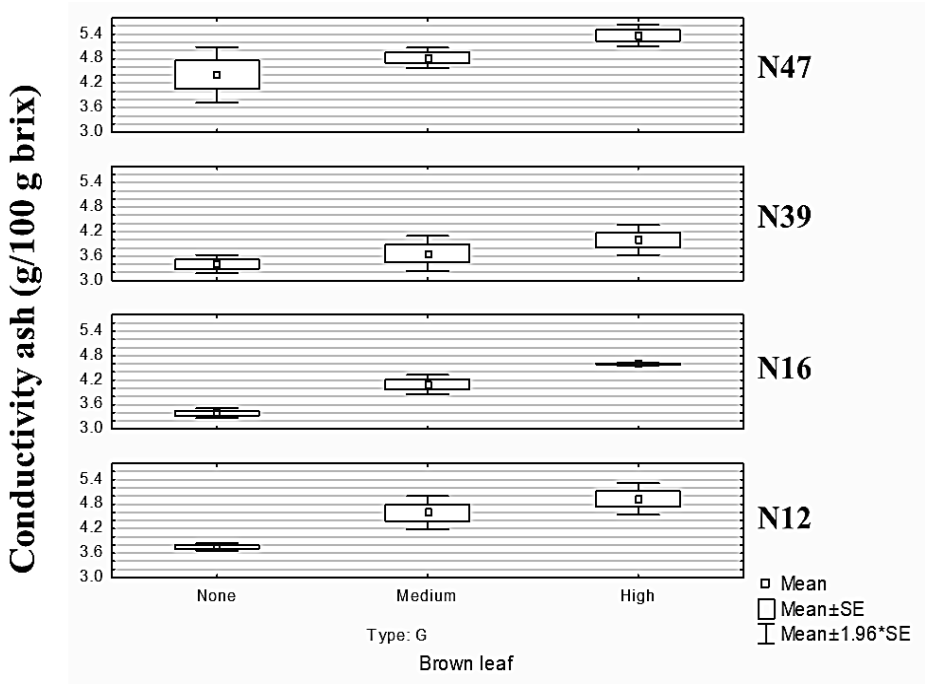

Figure 4-42: Box and whisker plot showing the comparison of conductivity ash content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

Rs: ash ratios (Table 4-7 and Figure 4-43) showed no statistically significant different effects since the reducing sugar results were not conclusive.

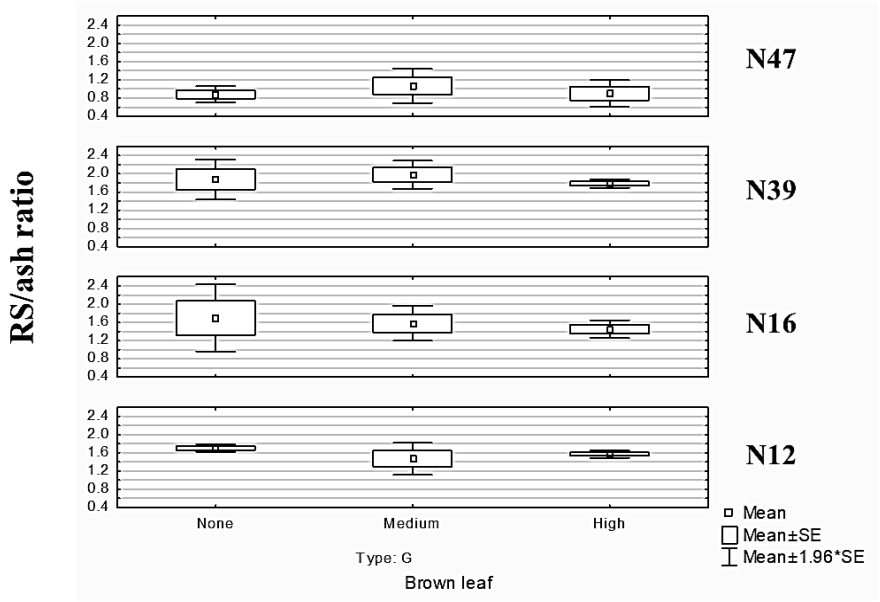

Figure 4-43: Box and whisker plot showing the comparison of reducing sugar: ash ratio for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For non-sucrose content, statistically significant differences were noted for most levels for almost all varieties (Table 4-7). N39 did not show statistically significant differences for two levels, none to medium and medium to high. Once again, this could be attributed to the deterioration of the variety, resulting in inaccurate results as discussed with the gravity purity results. Overall, the results showed that an increase in brown leaf content adds to non-sucrose content in extracted juices (Figure 4-44). The result is expected and can be related to the purity results.

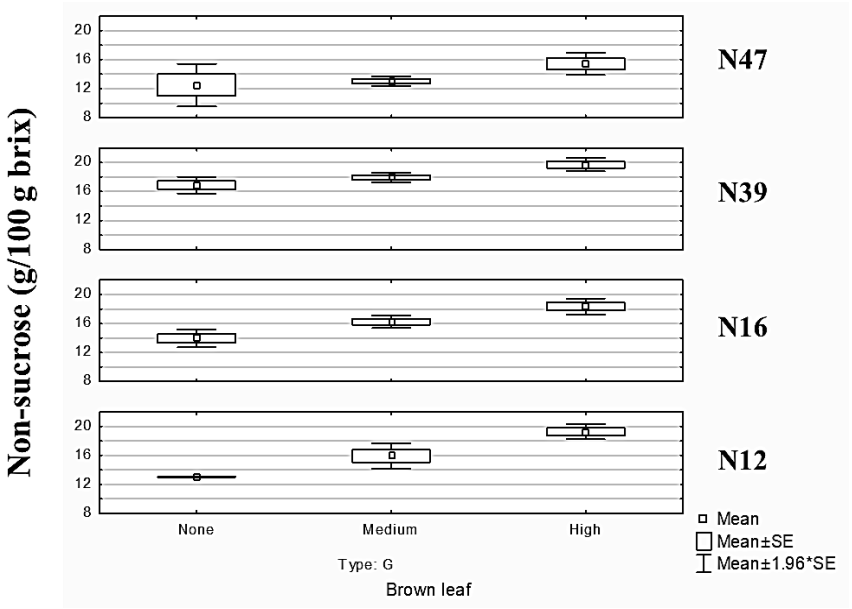

Figure 4-44: Box and whisker plot showing the comparison of non-sucrose content for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For sugarcane density, statistically significant differences were observed across all varieties at all levels of brown leaf (Table 4-7). It was found that the sugarcane density decreases with an increase in brown leaf content across all varieties (Figure 4-45) except with N16. The overall result is expected since the presence of brown leaf is believed to reduce the sugarcane density due to the fibrous nature of the brown leaf (Barker, 2017b). The odd results observed with N16 is suspected to be a possible error that occurred with either the samples or recording of data since a medium level of brown leaf showed a lower density than with a high level of brown leaf which is the opposite effect. This was confirmed by observing odd results with the moistures that was measured for the same samples.

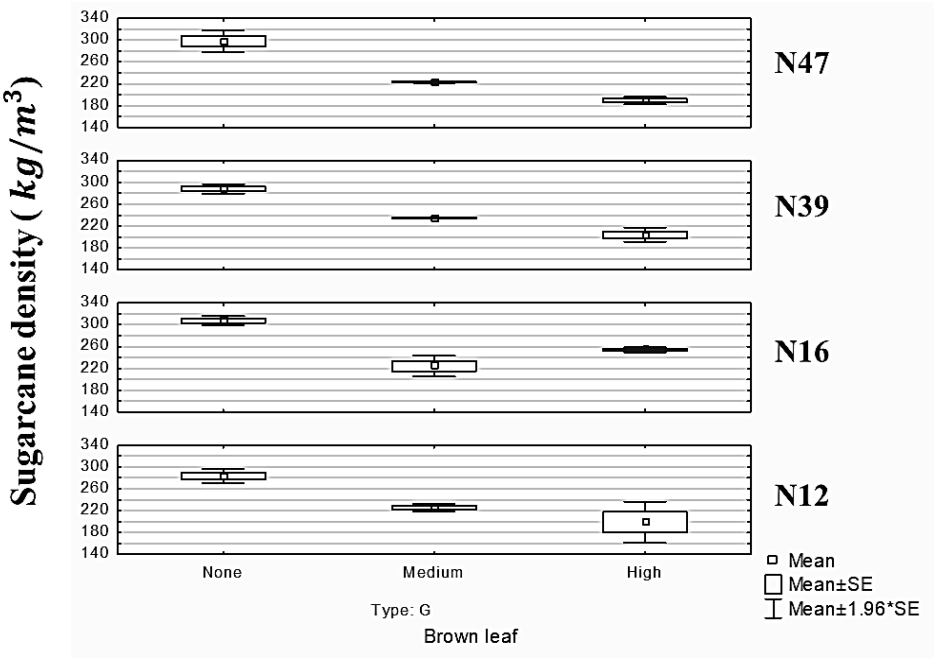

Figure 4-45: Box and whisker plot showing the comparison of sugarcane density for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

For percolation rates, statistically significant differences were found for only a few varieties (Table 4-7) namely N12 (none to medium and none to high) and N47 (none to high). For these two varieties, it was found that the percolation rate decreases with an increase in brown leaf content. The result was not expected since percolation tests conducted in a previous study (Loubser and Barker, 2011) reported the opposite effect where the percolation rates increased with the addition of brown leaf. However, it was considered that the preparation of the sugarcane samples used in this study was different to the preparation methods used in previous studies and the industry, hence this may have contributed to the unexpected result. Sugarcane prepared using a Waddell shredder as in this study is believed to be finer as compared to sugarcane prepared using industrial factory shredders as used by Loubser and Barker (2011). A picture showing the pronounced difference can be referred to in Appendix C: Figure C-1. It is assumed that the brown leaf prepared by the Waddell could also be absorbing some of the water, which could also contribute to the reduced percolation rates. However, further investigations must be undertaken to ascertain such assumptions.

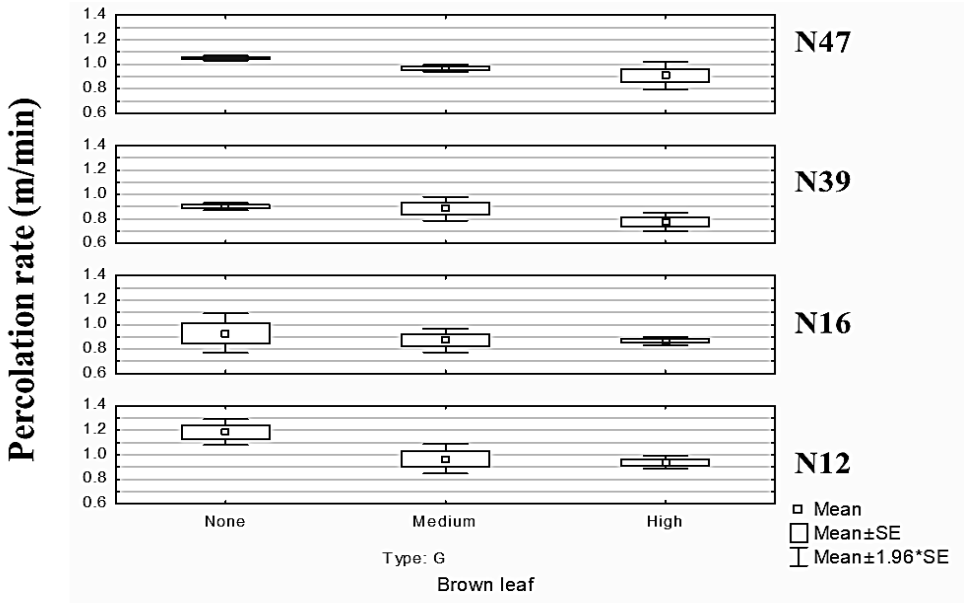

Figure 4-46: Box and whisker plot showing the comparison of percolation rate for extracted juices for different sugarcane varieties for green and burnt sugarcane. GN - green sugarcane with no brown leaf, B – burnt sugarcane, SE – standard error

4.3.3.3 Correlation coefficients and equations for green sugarcane with brown leaf for all varieties for all responses.

Table 4-8 shows the summary of the results for the regression plots that were drawn in TIBCO Statistica™ to determine the correlations for the % brown leaf contribution (X) per a response (Y) for each sugarcane variety and to determine a quantitative effect of brown leaf addition on each of the analytes present in the juice. Correlations are shown for each sugarcane variety since the sugarcane varieties are significantly different from each other in terms of characteristics (i.e. high colour variety etc.) and as a result, are expected to behave differently. Generalised model equations were initially considered to represent all four sugarcane varieties for each response, however, the model accuracy for the individual sugarcane varieties were much higher.

The scatterplots were plotted at a 95% confidence interval and the correlations were based on a linear fit (Appendix C). The p-values are also indicated to show the statistical significance of the results.

From the correlations in Table 4-8, approximately a 0.2-0.5% purity drop for every 1% increase in brown leaf content can be expected. Muir *et al.* (2009) conducted similar tests and reported 0.4% decrease in mixed juice purity for every 1% increase in leaf (green and brown) which agrees with the results obtained in this study.

For every 1% increase in brown leaf content, an approximate colour increase between 1300-1900 ICUMSA units can be expected. However, one needs to bear in mind that percentage colour increases will also depend on the colour of the particular sugarcane variety. Reid and Lionnet (1989) performed factory trials (on a milling tandem) and showed that increasing the fibre of the sugarcane with tops and brown leaf by 1% will increase the colour of mixed juice by 1841 IU. The factory trial used burnt sugarcane as the control and these tests were only conducted once but falls within the range of the results in this study.

Scott et al. (1978) showed that a 1% increase in brown leaf is likely to increase colour by 3.6%. This figure is much lower than for the investigations from this study, where a colour increase of between 6% and 12% for every percentage increase in brown leaf content of sugarcane were measured. However, Scott et al. (1978) used a sugarcane press to extract juices and this has been proven not to simulate extraction in a diffuser. Using cold digestion to extract the juice from sugarcane, Lionnet (1992) reported that the leaf component's (green and brown) are likely to increase the colour of sugarcane by between 4 and 15% per percentage increase in leaf and this is close to the range found in this study.

It can be expected that for every 1% increase in brown leaf content, a 1.2-2.4% increase in conductivity ash content can be expected. Scott et al. (1978) reported a 2.5% increase in sulphated ash and Lionnet (1982) reported a 2.4% increase in sulphated ash content for every 1% increase in brown leaf content for their investigations. Extractions were done using a sugarcane press.

For every 1 % increase in brown leaf content, an approximate increase between 0.2-0.4% in non-sucrose content can be expected. It is expected that for every 1% increase in brown leaf, an approximate decrease between 1.2-2.5% in sugarcane density is expected. The results tie in with work done by Barker (2017b) where tests were performed on factory - prepared sugarcane and it was estimated that the brown leaf content of the green sugarcane was about

15% (g/100 g sample). Barker (2017b) found that on average there was a 40% decrease in the sugarcane density.

A 1% increase in brown leaf content is expected to result in decreases between 0.5-1.4% in percolation rates. As discussed earlier in the box and whisker plots, the result was unexpected and did not agree with the work previously done in this area.

Table 4-8: Correlation coefficients and respective equations for X (% brown leaf) for each response (Y)

Variety N12			
Responses (Y)	**P -value**	**R-squared value**	**Equation**
Gravity purity %	0.0001	0.8971	Y=87.0212-0.4141X
Colour (IU)	0.0000	0.9299	Y=22875.3333+1474.4889X
Fructose/Brix (g/100 g sample)	0.5503	0.3028	Y=3.0456+0.0457X
Glucose/Brix (g/100 g sample)	0.2075	0.2160	Y=3.258+0.0434X
Conductivity ash/Brix (g/100 g sample)	0.0020	0.7671	Y=3.835+0.0784X
RS/ash ratio (g/100 g sample)	0.4390	0.0877	Y=1.6462-0.0088X
Non-sucrose/brix(g/100 g sample)	0.0001	0.8971	Y=12.9788+0.4142X
Sugarcane density (kg/m^3)	0.0016	0.7795	Y=278.1944-5.62X
Percolation rate (m/min)	0.0149	0.5954	Y=1.1522-0.0164X
Variety N16			
Responses (Y)	**P -value**	**R-squared value**	**Equation**
Gravity purity %	0.0005	0.8424	Y=86.02228-0.2936X
Colour (IU)	0.0001	0.8916	Y=16547.5+1869.4889X
Fructose/Brix (g/100 g sample)	0.5862	0.0444	Y=3.0368+0.0219X
Glucose/Brix (g/100 g sample)	0.3362	0.1321	Y=2.741+0.0425X
Conductivity ash/Brix (g/100 g sample)	0.00001	0.9429	Y=3.4199+0.0806X
RS/ash ratio (g/100 g sample)	0.4791	0.0739	Y=1.6986-0.0164X
Non-sucrose/brix(g/100 g sample)	0.0005	0.8424	Y=13.9772+0.2936X
Sugarcane density (kg/m^3)	0.0786	0.3769	Y=288.6444-3.5511X
Percolation rate (m/min)	0.4226	0.0939	Y=0.9206-0.0042X

Variety N39			
Responses (Y)	**P -value**	**R-squared value**	**Equation**
Gravity purity %	0.00330	0.7317	Y=83.2419-0.1883X
Colour (IU)	0.00000	0.9709	Y=14605.9444+1294.2444X
Fructose/Brix (g/100 g sample)	0.79680	0.0101	Y=3.8524+0.0292X
Glucose/Brix (g/100 g sample)	0.34290	0.1288	Y=3.2174+0.023X
Conductivity ash/Brix (g/100 g sample)	0.03950	0.4768	Y=3.3893+0.0395X
RS/ash ratio (g/100 g sample)	0.69110	0.0239	Y=1.9245-0.006X
Non-sucrose/brix(g/100 g sample)	0.00330	0.7317	Y=16.7581+0.1883X
Sugarcane density (kg/m^3)	0.00001	0.9431	Y=284.5611-5.6067X
Percolation rate (m/min)	0.05190	0.4388	Y=0.915-0.0082X
Variety N47			
Responses (Y)	**P -value**	**R-squared value**	**Equation**
Gravity purity %	0.06830	0.3985	Y=87.8202-0.197X
Colour (IU)	0.00000	0.9728	Y=11835.1667+1397.0889X
Fructose/Brix (g/100 g sample)	0.60510	0.0402	Y=2.1237+0.0259X
Glucose/Brix (g/100 g sample)	0.35550	0.1227	Y=2.0871+0.0332X
Conductivity ash/Brix (g/100 g sample)	0.01480	0.5958	Y=4.3849+0.0642
RS/ash ratio (g/100 g sample)	0.92120	0.0015	Y=0.9397+0.0014
Non-sucrose/brix(g/100 g sample)	0.06830	0.3985	Y=12.1798+0.197X
Sugarcane density (kg/m^3)	0.00004	0.9202	Y=291.2167-7.2378X
Percolation rate (m/min)	0.01500	0.5947	Y=1.0461-0.0096X

Overall, the correlations for the colour in the extracted juices showed very strong correlations and the best correlations from all responses tested across all varieties tested, indicating that the addition of brown leaf adds colour more than any other components to extracted juices.

Gravity purity and non-sucrose showed strong correlations for all sugarcane varieties except N47. For this variety, the three replicates for the juices that were extracted from sugarcane with no brown leaf had large deviations in gravity purity and this could have affected the correlation. It is also possible that this was due to the large variability in the sugarcane stalks as some were noted to be very thick and this may have caused scatter in the results and consequently poor correlations. The correlations for the conductivity ash results were moderately high which was acceptable considering the concerns regarding the analytical method. Correlations for the sugarcane density were strong across all varieties except for N16. Similarly, correlations for the percolation rates were moderately high for all varieties except N16. Reducing sugars and as a result, reducing sugar: ash ratios showed the poorest correlations, which relates to the reasoning around the dilution, analytical method used and the lower concentrations present.

The objectives of the brown leaf trials were achieved since valid conclusions could be drawn on the differences in juices and sugarcane density that was extracted from burnt and green sugarcane. In addition, differences in juice quality and sugarcane density could also be established from varying quantities of brown leaf. The results showed that juices extracted from green and burnt sugarcane differed in quality, more specifically for the colour, purity and impurity contents. The result with the percolation rates must be investigated further to draw sound conclusions and validate all assumptions made. Overall, it was also evident that brown leaf addition will result in lower juice purities and higher juice impurities.

5. CONCLUSIONS AND RECOMMENDATIONS

A pilot juice extractor technique was developed to extract a juice from sugarcane with a similar quality to juice extracted from a commercial diffuser. The pilot juice extractor was commissioned and subsequently validated against the GH sugar factory full-scale diffuser for burnt sugarcane. Attempts were made to also validate the pilot juice extractor for green sugarcane samples, but circumstances at the factory did not permit enough samples to draw conclusive results. The quality of the juice obtained from the pilot extractor was comparable to that of the draft juice obtained from the diffuser. The pilot juice extractor was used to investigate the effects of processing green sugarcane with varying quantities of brown leaf on the extracted juice quality. The effects on percolation rates were tested using an existing percolation column.

The commissioning tests highlighted that the pilot juice extractor technique was a suitable method be used for experimentation in assessing changes in diffuser juice quality, since the pilot juice extractor enabled the differentiation between juice extracted from sugarcane with and without brown leaf with significant differences noted for juice quality parameters such as colour, glucose and fructose concentrations. The results from the commissioning tests also indicated that the pilot juice extractor juice quality was significantly different (in most cases found between the two extremes) to the juice extracted from the cold digester and press methods. Establishing suitable operating conditions of contact time and temperature for the pilot extractor based on their statistical significance on the juice quality parameters was achieved and considerable recommendations were made for operation during the validation phase. An operating time of 30 minutes was recommended to maximise the number of tests per day and a temperature of 80°C was selected on the basis of the review of diffuser related pilot work.

The validation phase revealed that the pilot extractor juice quality showed better agreement with the quality of draft juice than for the DAC extract and press juices. The tests highlighted limitations of the study such as; large degrees of mixing which exist in the diffuser making it difficult to correlate each sugarcane consignment to its draft juice and that the diffuser and preparation equipment on the diffuser line were not optimized and configured to process green sugarcane. Furthermore, as mentioned earlier, there exists some notable differences between the pilot juice extractor and the diffuser. These limitations are possible reasons as

to why the pilot extractor juice did not show very strong correlations with draft juice although it showed the best correlations out of the three tested methods. The extractor juice showed much higher levels of conductivity ash than the DAC extract and press juice. It is thought that this is due to the analytical method of determining ash and for future work, it is highly recommended to budget for and consider alternative methods.

The final stage of the project showed that juices extracted from different sugarcane varieties showed varying colour and purity concentrations. The study revealed that an increase in brown leaf content had a directly proportional relationship with colour and conductivity ash. The increase in brown leaf content had an inversely proportional relationship with gravity purity, sugarcane density and percolation rate. It is recommended that the effect of brown leaf addition on percolation rates be investigated further, due to the unexpected results found in this study and that sulphated ash be considered as an alternative analytical method to conductivity ash for measurement of ash content in the juices in future work.

It may be argued that validation of the pilot juice extractor is required for green sugarcane. For this to happen, a sugar factory must be sourced to process simultaneous consignments of green sugarcane through their diffuser. However, the results from this study has indicated that nevertheless, perfect correlations can never be achieved due to the complexity and configuration of the existing sugarcane diffusers in sugar mills. Performing such work would not provide any more value to the findings and learnings that have already come out of this study. It is therefore recommended that the pilot juice extractor be used as a tool to investigate the effects of co-processing green sugarcane with brown leaf in commercial diffusers by using sugarcane varieties specific to various sugar factories. Such experiments will allow for information to be gathered on how each sugarcane variety would impact on the extracted juice quality and the process as a whole, since this study has only focused on four sugarcane varieties. This will allow for each factory to make a sound decision on whether co-processing green sugarcane would be a viable option or whether the green sugarcane would need to be de-leafed before being processed, as opposed to the current harvesting method of burning. Once the effects on juice quality and the processes are known per a variety for specific factories, a techno-economic study of the two alternative methods is recommended before deciding whether to co-process or remove brown leaf prior to processing if sugarcane burning is prohibited in the near future.

6. REFERENCES

Ahmed, J. and Rahman, M. S. 2012. *Handbook of food process design*. United States: Wiley-Blackwell.

American laboratory. 2019. *HPLC-PAD*. Available: https://www.americanlaboratory.com/914-application-Application-Notes/35296-The-Power-of-Pulsed-Amperometric-Detection-Coupled-With-Chromatography-for-Analyzing-Carbohydrates (Accessed 20 July 2018).

Barker, B. and Davis, S. 2010. Audit of extraction plant at Gledhow (RC). Durban: Sugar Milling Research Institute.

Barker, B. 2017a. *Effect of crushing green cane on factory operations*. Durban: Sugar Milling Research Institute.

Barker, B. 2017b. *Percolation tests with green cane at Ubombo factory*. Durban: Sugar Milling Research Institute.

Berk, Z. 2013. *Food process engineering and technology*. 2nd edition ed. Netherlands: Elsievier.

Bernhardt, H. W. and Pillay, V. 2000. Impacts of green cane harvesting on sugar factory operation at Sezela. In: Proceedings of *South African Sugar Technologists Association*. Kwa Shukela, Durban, 30 July-2 August 2000. Durban: Sugar Milling Research institute, 369-372.

Boote, G. L. N. 2010. A review of the cause and effects relationships within the processes of a sugar factory. MSc Agricultural Engineering, University of KwaZulu-Natal.

Buchanan, E. J. 1967. *Direct sampling and analysis of individual cane consignments. Part 1: Rapid cane and bagasse analysis using the SMRI cold extractor*. Durban: Sugar Milling Research Institute.

Buchanan, E. J. and Julliene, L. M. S. A. 1969. *Some observations on diffusion of cane percolation and submerged techniques*. Durban: Sugar Milling Research Institute.

Buchanan, E. J. and Brokensha, M. A. 1974. The application of direct cane testing to the south African sugar industry. In: Proceedings of International Society of Sugar Cane Technologists. Durban, 13-29 June 1974. 1456-1468.

Ceutics. 2014. HPLC versus GC. Available: https://www.slideshare.net/mobile/ceutics1315/hplc-vs-gc (Accessed 20 July 2018).

Costa, M. P. and Conte-Junior. 2015. Chromatographic methods for the determination of carbohydrates and organic acids in foods of animal origin. *Comprehensive reviews in food science*, 14: 586-600.

Davis, S. B. 1996 *The results of tracer tests on the Umfolozi diffuser*. Durban: Sugar Milling Research Institute.

Davis, S. B. and Barker, B. 2013. Investigations into alternative bagasse analysis methods. Durban: Sugar Milling Research Institute.

Eggleston, G., Grisham, M., Trew, T., Triche, R. and Antoine, A. 2009. Potential biomass quantity and sugar processing quality of trash and stalk tissues by different US sugarcane varieties. 111: 108-118.

Ghada, A., Rahman, A. and Razig, E. S. 2009. Evaluation of sugarcane juice quality as influenced by cane treatment and separn concentrations. Master's in science and Agriculture, University of Khartoum.

Gómez, E. O., Souza, R. T. G. D., Jackson, G., Rocha, D. M., Almeida, E. D. and Cortez, L. A. B. 2014. *Sugarcane trash as feedstock for second generation processes.* São Paulo: Editora Edgard Blücher.

Govindaraj, R. and Sankaranarayanan, P. 1996. On-line estimation of colour, turbidity and pH values in sugar refining processes. *Indian Journal of Engineering and Material Sciences*, 3: 96-100.

Graham, W. A., Morris, R. M. and Oosthuizen, D. M. 1968. Preliminary physiochemical studies on sugarcane diffusers. In: Proceedings of International Society of Sugar Cane Technologists. Taipei, Taiwan, 4-10 March 1968. 122-131.

Hemaida, S. E. A., Sayed, G. E. K. and El-Badawi, A. A. 1977. Effect of green cane and dry trash on cane and milling qualities. In: Proceedings of International Society of Sugar Cane Technologists. Sao Paulo, Brazil, 9-25 September 1977. Egypt: Kom-Ombu Cane Research Station of the Egyption Sugar and Distillation Company, 2485-2492.

Hugot, E. 1986. *Handbook of cane sugar engineering*. 3rd ed. New York: Elsevier.

Jensen, P. 2013. Continuous percolation rate measurement in a sugarcane diffuser. In: Proceedings of *South African Sugar Technologists' Association*. Durban ICC, 6-8 August 2013. 402-421.

Just chromatography. 2012. *High performance liquid chromatography*. Available: www.justchromatography.com/chromatography/hplc (Accessed 19 July 2018).

Kent, G. A. 2007. The effect of trash on the operation and performance of a raw sugar factory. In: Proceedings of *International Society of Sugar Cane Technologists"*. Durban,30 July 2007. Queensland university of technology, 1622-1628.

Kumar, V., Kumari, O., Singh, S., Upadhyay, R., Kumar, S. and Tripathi, M. 2012. Significance of Conductometric Analysis in Sugar Industry. *International Journal of Chemical and Analytical Science*, 3 (3).

Leal, M. R. L. V. and Hassuani, S. J. 2000. The collection of sugarcane bagasse and trash for an advanced cogeneration system. *International Society of Sugar Cane Technologists' Workshop*, Mauritius.

Legendre, B. L., Clarke, M. A., Godshell, M. A. and Grisham, M. P. 1999. Developments in sugarcane agriculture that affect processing. *Zuckerindustrie*, 124 (2): 120-125.

Lionnet, G. R. E. 1985. A study of the extraction of non-sucrose components of sugarcane (Saccharum officinarum). Master of Science, University of Natal.

Lionnet, G. R. E. 1989. *A survey of diffuser operating conditions and performances*. Durban: Sugar Milling Research Institute.

Lionnet, G. R. E. 1996. *The effects of some cane characteristics on extraction and dewatering*. Durban: Sugar Milling Research Institute.

Lionnet, G. R. E., Pillay, M. and Thibela, B. 2005. The effects of selected factors on percolation in pilot diffusion columns. In: Proceedings of *South African Sugar Technologists' Association*. Durban 19-22 July 2005. Durban: Sugar Milling Research Institute.249-256.

Loubser, R. C. and Barker, B. 2011. Cane characterisation: The percolation test. In: Proceedings of *South African Sugar Technologists' Association*. Durban ICC, 17-19 August 2011. Durban: Sugar Milling Research Institute. 413-422.

Love, D. J. and Rein, P. W. 1980. Percolation behaviour of a cane diffuser. In: Proceedings of International Society of Sugar Cane Technologists Manilla, Phillipines, 1-10 February 1980. 1900-1924.

Lund, A. and Lund, M. 2018. *Pearson Product-Moment Correlation*. Available: statistics.laerd.com/ (Accessed 20 January 2019).

Madho, S., Davis, S. and Bhyrodeyal, B. 2017. Ninety-second annual review of the milling season in southern Africa (2016-2017). In: Proceedings of *South African Sugar Technologists' Association*. Durban ICC, 15-17 August. Durban: 20 - 25.

Menandro, L. M. S., Cantarella, H., Franco, H. C. J., Kolln, O. T., Pimenta, M. T. B., Sanches, G., Rabelo, S. C. and Carvalho, J. L. N. 2017. *Comprehensive assessment of sugarcane straw: implications for biomass and bioenergy production*. Available: https://onlinelibrary.wiley.com/doi/10.1002/bbb.1760/full%20Go%20to%20publication%20Download (Accessed 29 May 2018).

Ming, O. S. 2007. Comparative study of optimisation of continuous counter-current extraction licorice. Master of Science, National University of Singapore.

Muir, B. and Eggleston, G. 2009 *The effect of green sugarcane on MJ quality at Felixton*. Sugar Milling Research Institute.

Muir, B. M., Eggleston, G. and Barker, B. 2009. *The effect of green sugarcane on downstream processing*. Durban: Sugar Milling Research Institute

Mullapudi, N. 2010. Comparison of Diffusion and Milling at a Cane Sugar Plant. In: Proceedings of *International Society of Sugar Cane Technologists*. Venacruz, Mexico, 7-11 March 2010. Tanuku, India: The Andhra Sugars Ltd, 1-9.

Pamu, V. 2012. Effect of different diffusion strategies on sugar extraction from sweet sorghum. Master of Science, Oklahoma State University.

Parfitt, R. C. 2005. Release of sugarcane varities in South Africa. In: Proceedings of *South African Sugar Technologists' Association*. Durban, 19-22 August 2005. Durban: SASRI, 63-71.

Payne, J. H. 1968. Cane diffusion - the displacement process in principle and practice. In: Proceedings of International Society of Sugar Cane Technologists. Taipei, Taiwan, 4-10 March 1968. 103-121.

Perten. 2019. *Near Infrared Spectroscopy*. Available: http://www.perten.com/publications/articles/NIR-Introduction1/ (Accessed 19 July 2018).

Pol, C. V. D. 1957. *Some notes on the extraction of sucrose from cane by diffusion*. Durban: Sugar Milling Research Institute.

Prabhakar, N., Raju, D. V. L. N. and Sagar, R. V. 2010. Cane trash as fuel. Paper presented at the *International Society of Sugarcane Technologists"*. Venacruz, Mexico, 7-11 March 2010. Nava Bharat Ventures LTD, 1-11.

Rama, S., Dehrman, R. A., Zungu, H. and Sweet, D. G. 2006. The effect of clay type soil in the diffuser at Umfolozi mill. In: Proceedings of *South African Sugar Technologists' Association*. Durban ICC, 18-20 July 2006. Durban: Ushukela Milling, 320-326.

Reid, M. J. and Lionnet, G. R. E. 1989. The effects of tops and trash on cane milling based on trials at Maidstone. In: Proceedings of *South African Sugar Technologists' Association*. South African Sugar Experimentation Station, Durban, 5-8 June. Durban: Sugar Milling Research Institute
3-6.

Rein, P. W. 1972. A survey study of the cane sugar diffusion process. PHD, University of Natal.

Rein, P. W. 2007. Cane sugar engineering. 1st ed. Berlin, Germany: Bartens.

Rein, P. W. and Ingham, P. J. S. 1992. Diffuser performance optimisation through control of liquid flow patterns. In: Proceedings of International Society of Sugar Cane Technologists. Bangkok, Thailand, 5-14 March 1992. 779-796.

Saska, M., Zossi, B. S. and Liu, H. 2010. Removal of colour in sugar cane juice by clarification, defecation, sulfitation and carbonation. In: Proceedings of *International Society of Sugarcane Technologists'*. Venacruz, Mexico, May 2010. Louisiana, USA Audubon Sugar Institute, 1-14.

SASTA. 2009. SASTA Laboratory Manual including the Official Methods. 5th edition ed. Mount Edgecombe, South Africa: South African Sugar Technologists' Association.

Scott, R. P., Falconer, D. and Lionnet, G. R. E. 1978. A laboratory investigation of the effects of tops and trash on extraction, juice quality and clarification. In: Proceedings of *South African Sugar Technologists' Association*. South African Sugar Experimentation Station, Durban, 5-9 June 1978. Durban: Hulett Sugar Limited, 51-53.

Seader, J. D., Henley, E. J. and Roper, D. K. 1998 *Separation process principles: Chemical and biochemical operations*. 3rd Edition ed. New York, United states: John Wiley incorporated.

SMRI. 1978. Colloquium on diffusion. Durban, South Africa: Sugar Milling Research Institute.

SMRI. 2011. *Determination of glucose, fructose and sucrose in cane juices, syrups and molasses by high performance liquid chromatography*, ICUMSA method G57/8/4.Durban, South Africa: Sugar Milling Research Institute.

SMRI. 2012. Essential sugar technology. Sugar Milling Research Institute. Durban, Kwa-Zulu Natal. March 2012.

SMRI. 2013. *Determination of the colour in mixed juice*, TM 021. Durban: Sugar Milling Research Institute.

SMRI. 2014. *The analysis of mixed juice composite samples by Near-infrared spectroscopy*, TM 401. Durban: Sugar Milling Research Institute.

SMRI. 2018a. *The determination of conductivity ash in juice and molasses*, TM 066. Durban: Sugar Milling Research Institute.

SMRI. 2018b. *The determination of glucose, fructose and sucrose in cane mixed juice by Gas chromatography*, TM 300. Durban: Sugar Milling Research Institute.

SMRI. 2018c. *Determination of refractometer brix in juice*, TM 005. Durban: Sugar Milling Research Institute.

Statistics Canada. 2017. *Constructing box and whisker plots*. Available: www.statcan.gc.ca (Accessed 20 January 2018).

Statisticshowto. 2014. *ANOVA Test: Definition, Types, Examples*. Available: statisticshowto.datasciencecentral.com (Accessed 20 February 2019).

Tarique, S. M. 2018. *Colour development*. Available: http://www.psst.org.pk/48Convention/03-COLOUR%20DEVELOPMENT%20AND%20REMOVAL,%20By%20Syed%20M.Tariq%20&%20Sharif%20Khan.pdf (Accessed 28 May 2018).

Thai, C. and Doherty, W. 2011. The composition of sugarcane juices derived from burnt cane and whole green cane crop. In: Proceedings of *The Australian Society of Sugar Cane Technologists'*. Mackay Entertainment and convention centre, 4-6 May 2011. Brisbane Sugar Research and Innovation Centre of Tropical Crops and Bio Commodities-Queensland University of Technology, 1-9.

Van Antwerpen, R. 2010. *The pros and cons of trashing or burning at harvest*. Available: https://sasri.org.za/storage/Information_Sheets/IS_4.7-Trashing-or-burning-at-harvest.pdf (Accessed 20 January 2019).

Venugopal, K. 2017. *Report on process overview of Gobind Sugar Mills LTD*. Adventz Group. Available:www.slideshare.net/ ashish 23993/ (Accessed 20 August 2019).

Walford, S. N. 1996. Composition of cane juice. In: Proceedings of *South African Sugar Technologists' Association*. South African Sugar Experimentation Station, Durban, 3-6 June. Durban: Sugar Milling Research Institute. 265-266.

Walford, S. N., Schaffler, K. J. and Boil, P. G. M. D. 2004. Analytical chromatographic solutions for sugar processing. In: Proceedings of *Proceedings of the South African Sugar Technologists' Association*. Durbann ICC, 27-30 July 2004. Durban: Sugar Milling Research Institute, 505-522.

Walsh, G. 1998. The Riviere juice extractor. In: Proceedings of *South African Sugar Technologists' Association*. Durban ICC, 1-3 June 1998. Technoserve CC, 34-42.

Wienese, A. 2002. *Plant installations 200*. Durban: Sugar Milling Research Institute.

A Appendices

A1. Viability and verification of pilot juice extractor

Table A-1: Mass of sugarcane constituents

Variety	N12	N19
Mass of tops (kg)	29.00	21.36
Mass of clean sugarcane stalks (kg)	101.14	112.33
Mass of brown leaves (kg)	12.24	21.40
Total mass (kg)	142.38	155.09
% contribution of clean sugarcane stalk	71.04	72.43
% contribution of brown leaf	8.60	13.80

Table A-2: Two factorial experimental design for 18 combinations of operating conditions for the pilot juice extractor

Test number	Replicates	Centre point	With brown leaf	Time (minutes)	Temperature (°C)
15	3	1	No	30	85
5	2	0	No	45	80
16	2	1	Yes	60	85
9	2	1	No	30	85
3	2	1	Yes	30	75
12	1	0	Yes	45	80
13	1	1	No	30	85
14	1	1	No	60	75
8	2	1	No	60	75
1	1	1	Yes	30	75
11	3	0	No	45	80
6	2	0	Yes	45	80
17	1	0	No	45	80
10	3	1	Yes	60	85
2	3	1	No	60	75
7	3	1	Yes	30	75
18	3	0	Yes	45	80
4	1	1	Yes	60	85

Table A-3: Results for the pilot juice extractor for the commissioning trials

Test	Brown leaf	Time (minutes)	Temp (°C)	Fructose (g/100 g brix)	Glucose (g/100 g brix)	Gravity purity (%)	Cond ash (g/100 g brix)	Colour (IU)	RS/ash ratio
15	No	30	85	0.84	0.73	88.85	2.79	16509	1.57
5	No	45	80	0.67	0.70	91.58	3.51	16586	1.37
16	Yes	60	85	0.76	0.83	89.44	1.98	36788	1.58
9	No	30	85	0.74	0.70	93.33	2.81	23371	1.44
3	Yes	30	75	0.87	0.84	81.82	3.50	32948	1.71
12	Yes	45	80	0.73	0.80	88.89	3.47	32552	1.53
3	No	30	85	0.80	0.80	89.62	2.42	17028	1.59
14	No	60	75	0.62	0.52	91.38	3.10	16686	1.14
8	No	60	75	0.67	0.60	92.98	2.46	15455	1.26
1	Yes	30	75	0.77	0.74	88.07	3.16	33133	1.51
11	No	45	80	0.93	0.73	87.38	2.99	15821	1.66
6	Yes	45	80	0.93	0.87	88.24	2.77	42844	1.80
17	No	45	80	0.80	0.63	95.12	2.79	15198	1.43
10	Yes	60	85	0.85	0.68	86.44	2.71	44142	1.53
2	No	60	75	0.60	0.47	85.05	1.99	17131	1.06
7	Yes	30	75	1.20	0.99	82.53	2.40	37589	2.19
18	Yes	45	80	0.93	0.79	87.24	3.10	34648	1.72
4	Yes	60	85	0.92	0.86	78.29	2.63	43003	1.78

Table A-4: Results for the DAC extract and press juice for the commissioning trials

DAC										
Test no.	Brown leaf	Brix %	Fructose %	Glucose %	Reducing sugars (F +G)%	Gravity purity %	Cond ash %	Colour (IU)	Rs/ash	Moisture %
16	Yes	6.19	1.94	1.71	3.65	79.48	1.13	28691	3.23	61.42
1	Yes	6.12	2.96	2.04	5.00	88.24	2.94	29496	1.70	57.34
18	Yes	6.01	1.78	2.65	4.43	77.70	2.50	62805	1.77	59.81
9	No	6.37	2.72	2.12	4.84	89.48	2.67	13772	1.81	67.51
14	No	6.22	2.70	2.06	4.76	89.39	2.25	15661	2.11	69.07
2	No	6.45	2.43	2.19	4.62	85.12	2.02	17793	2.29	68.06
PRESS										
Test no.	Brown leaf	Brix %	Fructose %	Glucose %	Reducing sugars (F +G)%	Gravity purity %	Cond ash %	Colour (IU)	Rs/ash	Moisture %
16	Yes	23.36	0.45	0.51	0.97	91.14	2.65	10809	0.365	61.42
1	Yes	23.42	0.53	0.77	1.31	90.61	2.78	11565	0.471	57.34
18	Yes	23.44	0.68	0.46	1.13	90.10	2.90	15769	0.391	59.81
9	No	22.28	0.61	0.78	0.97	91.02	2.69	9474	0.359	67.51
14	No	21.11	0.61	0.80	1.40	90.72	2.89	10451	0.485	69.07
2	No	22.10	0.71	0.64	1.35	92.99	2.31	12737	0.584	68.06

B Validation

The tracer test

A lithium tracer was conducted on the GH diffuser front-end to determine the time at which most of the brix of the sugarcane is released into the juice from a specific sugarcane consignment.

The methodology used to conduct the lithium tracer test was according to Davis (1996). Approximately 8.5 kg of lithium chloride was required to give an average lithium concentration (approximately 50 mg/kg draft juice) in draft juice that would be easily detected by the lithium analysis. It was agreed that a lithium tracer would need to be added by first soaking enough shredded sugarcane with the lithium chloride solution and then emptying the buckets onto the main carrier feeding the diffuser. It was established that four buckets of shredded sugarcane (28 kg) would be sufficient to enable the tracer to be introduced across the full width of the diffuser. Tests done in the laboratory showed that a sugarcane to water ratio of 1:0.75 will allow all liquid to be absorbed by the sugarcane fibre. The lithium chloride was dissolved in 12.08 kg of water to deliver the required mass of lithium chloride solution (21 kg). The ratio of sugarcane to lithium chloride was 28:21 (m:m).

Following this procedure, shredded sugarcane was collected from the CTS sampling hatch and was soaked in the lithium chloride solution for a period of 15 minutes. After soaking, the four buckets of sugarcane sample were emptied onto the shredded sugarcane conveyor just ahead of the CTS sampling point. The draft juice was sampled simultaneously with the addition of the tracer. The draft juice was sampled at the draft juice pump every 30 seconds for 15 minutes, then every minute up to 30 minutes and thereafter sampled every 2 minutes until the total time of five times the calculated diffuser juice residence time was reached (approximately 100 minutes).

The bulk of the lithium tracer exited in the draft juice between nine to 10 minutes (Figure B-1) and is indicative that the bulk of the brix in the sugarcane, sampled at the CTS sampling hatch, would be expected to exit in the draft juice at the same residence time.

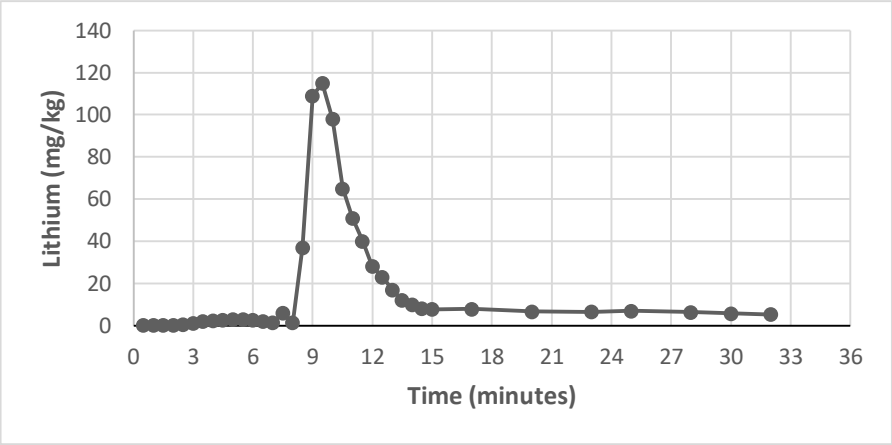

Figure B-1:Lithium tracer test graph showing peak lithium concentration in draft juice exiting the diffuser

Table B-1: Summary of sugarcane consignment information used in the validation

SMRI No.	Date	Time stamp of sugarcane sample	Variety	*B/G/L
Day 1 Sample 1	2018/10/23	11:10	N37	G+L
Day 1 Sample 2	2018/10/23	13:45	N39	B
Day 2 Sample 1	2018/10/25	10:45	N31	B
Day 2 Sample 2	2018/10/25	12:30	N31	B+L
Day 2 Sample 3	2018/10/25	14:10	N31	B
Day 3 Sample 1	2018/10/25	10:15	N16	B+L
Day 3 Sample 2	2018/10/25	11:50	N37	B+L
Day 3 Sample 3	2018/10/25	13:50	N12	B+L
Day 4 Sample 1	2018/10/30	10:45	N50	G+L
Day 4 Sample 2	2018/10/30	12:30	N16	B
Day 4 Sample 3	2018/10/30	14:05	N16	B
Day 5 Sample 1	2018/10/30	12:00	N39	B
Day 6 Sample 1	2018/10/31	10:45	N37	G+L
Day 7 Sample 1	2018/11/06	10:28	N50	G+L
Day 7 Sample 2	2018/11/06	12:08	N39	B
Day 7 Sample 3	2018/11/06	13:19	N39	B

*** G = green, B = burnt, L = various amount of leaf**

Correlation plots for fructose and glucose showing outliers observed for draft juice

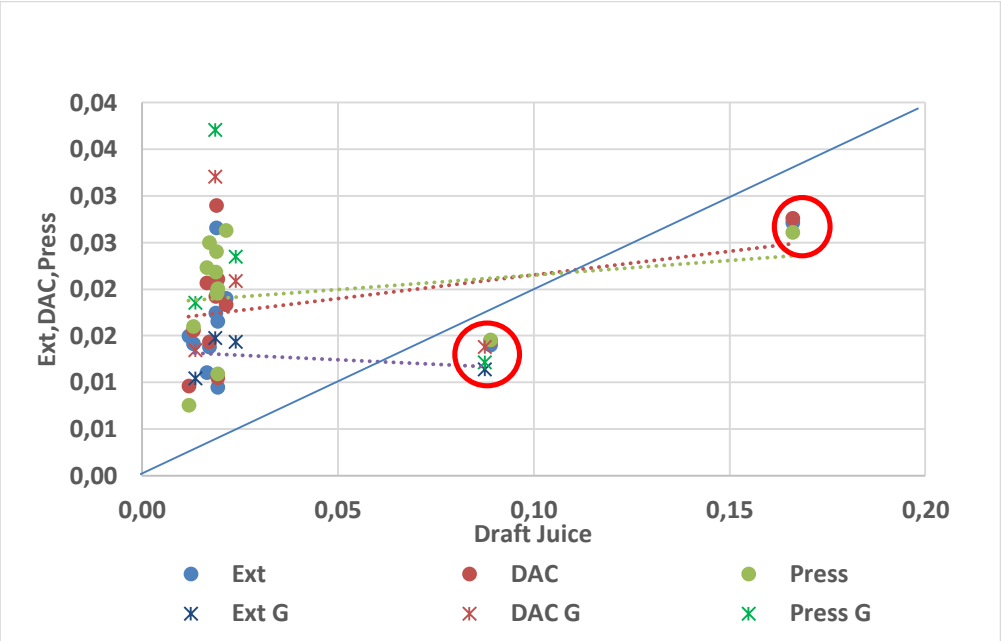

Figure B-2:Fructose (g/100g brix) correlations with outliers for extracted juices against draft juice with the equivalence line represented by a solid blue line

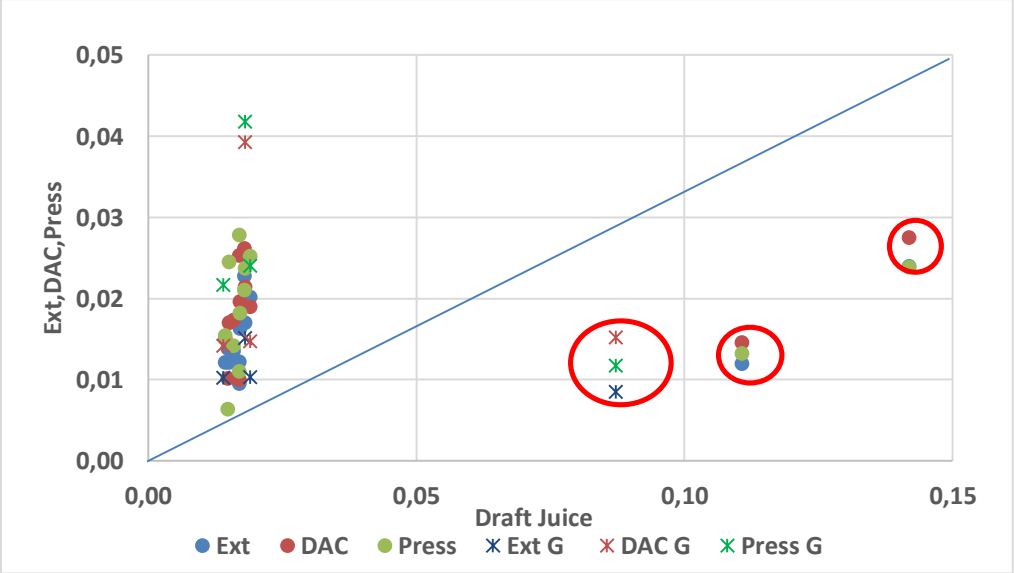

Figure B-3: Glucose (g/100g brix) correlations with outliers for extracted juices against draft juice with the equivalence line represented by a solid blue line

C Brown leaf experiments

Table C-1: Contribution of various components of the uncleaned sugarcane as per a variety

Variety	N12	N16	N39	N47
Mass of tops (kg)	9.88	3.44	9.64	5.12
Mass of clean sugarcane stalks (kg)	101.48	101.92	100.82	100.43
Mass of brown leaves (kg)	16.06	20.04	14.88	9.36
Total mass (kg)	127.42	125.40	125.34	127.42
% contribution of clean sugarcane stalk	79.64	81.28	80.44	79.60
% contribution of brown leaf	15.83	19.66	14.76	9.23

Table C-2: Details of sugarcane cutting, collection and preparation and purity deterioration

Sugarcane details				
Variety	**Date cut**	**Date burnt**	**Date collected and cleaned**	**Experimental test dates**
N12	6 Feb	7 Feb	7th & 8th Feb	11th ,12th and 13th Feb
N39	6 Feb	7 Feb	7th & 8th Feb	13th & 14th Feb
N47	6 Feb	7 Feb	7th & 8th Feb	15th and 16th Feb
N16	6 Feb	7 Feb	7th & 8th Feb	18th & 20th Feb
Purity deterioration checks				
Variety	**Control test date**		**Experimental test dates**	**Purity difference**
N12	11 Feb		11th ,12th and 13th Feb	0.00
N39	11 Feb		13th & 14th Feb	2.46
N47	11 Feb		15th and 16th Feb	-0.47
N16	11 Feb		18th & 20th Feb	0.74

Table C-3: Experimental design output from TIBCO™ Statistica

Test	Variety	Brown leaf	% Brown leaf		Test	Variety	Brown leaf	% Brown leaf
9	N12	Burnt	0.0		2	N39	Medium	7.5
10	N12	Medium	7.5		6	N39	Burnt	0.0
11	N12	High	15.0		8	N39	High	15.0
12	N12	Burnt	0.0		14	N39	Medium	7.5
27	N12	Medium	7.5		24	N39	None	0.0
30	N12	High	15.0		26	N39	None	0.0
33	N12	None	0.0		29	N39	Burnt	0.0
34	N12	None	0.0		32	N39	None	0.0
37	N12	Burnt	0.0		39	N39	High	15.0
38	N12	High	15.0		43	N39	High	15.0
40	N12	None	0.0		44	N39	Medium	7.5
47	N12	Medium	7.5		45	N39	Burnt	0.0
3	N16	Medium	7.5		1	N47	Medium	7.5
5	N16	Medium	7.5		4	N47	High	15.0
7	N16	Burnt	0.0		13	N47	High	15.0
15	N16	Burnt	0.0		16	N47	Medium	7.5
19	N16	High	15.0		17	N47	Medium	7.5
21	N16	None	0.0		18	N47	Burnt	0.0
23	N16	None	0.0		20	N47	High	15.0
25	N16	Burnt	0.0		22	N47	Burnt	0.0
28	N16	Medium	7.5		35	N47	Burnt	0.0
31	N16	High	15.0		36	N47	None	0.0
42	N16	High	15.0		41	N47	None	0.0
48	N16	None	0.0		46	N47	None	0.0

Table C-4: Raw data for cane density, percolation and moisture tests

Density data				
Height 1(cm)	Height 2(cm)	Height 3(cm)	Average height(cm)	
232	236	235	234,33	
229	229	231	229,67	
227	227	230	228,00	
Moisture data				
Initial mass(g)	Tray mass(g)	Final mass(g)		
1050,04	959,04	988,64		
785,71	685,71	715,68		
1060,17	960,17	988,80		
Percolation rate				
Time measured at (min)	mass(g)	Time(sec)	mag flow(l/min)	overflow(l/min)
20	521,08	10	34,98	2,17
25	560,27	10	34,89	2,40
30	582,08	10	34,79	2,53

Sample calculation for percolation rate, sugarcane density and moisture % for brown leaf trials for Test 40. The 1st set of the triplicate result is shown here.

Density calculation

Density = (mass of cane (kg))/ (volume)

= (mass of cane (kg))/ ((area x average height) m^3)

Average height = (height 1+height 2+height 3)/3

A = (232+236+235)/3

= 234.33 mm

Hence Area =A=πr^2

= π 〖(76mm) 〗 ^2

=18146 mm2

Mass of cane = 1.2kg

Density = (1.2 (kg))/ (18146 x 234.33 x 1/ (1 x 〖10〗 ^9))

=286.73 kg/m^3

Density apparatus diameter = 152mm

Percolation rate

Percolation rate = (Flow rate (m^3/ (min)))/(Area (m^2))

Net flow rate= mag flow rate(L)- overflow(L)

=34.98 - 2.17

=32.81L

Net Flow rate = mass(kg)/volume(L)

= 32.81 L/min = 0.03281 m3 /min

Area = πr^2

Diameter measured = 19cm

So r = 19/2 = 9.5cm = 0.95 m

Area = πr^2

= π {(0.95)} ^2

= 0,028353 m2

So percolation rate= (0.03281 m^3/min)/0,028353 m^2)

= 1.157 m^3/min

Moisture calculation

Final mass (g) = [Final mass(g)- drying tray mass(g)]

=1050.04 - 959,04

=91 g

Initial mass(g)= [Initial mass(g)- drying tray mass(g)]

=988,64 – 959,04

=29.6 g

Moisture (%) = 100 – [Final mass(g)/initial mass(g) x100]

=100 – [(29.6/91) x 100]

= 67.47

The final result presented in Table C-5 is achieved by performing all three triplicate calculations for each test and taking the average of the three tests.

Table C-5: Results from experimental trials for the pilot juice extractor

Test	Variety	Type	% Brown leaf	Brown leaf	Brix (g/100 g)	Gravity Purity (%)	Fructose (g/100 g Brix)	Glucose (g/100 g Brix)	Cond ash (g/100 g Brix)	Colour (IU)	RS/ash	Non-sucrose (g/100 g Brix)	Cane density (kg/m^3)	Moisture %	Perc rate (m/min)
1	N47	G	7.5	Medium	2.55	87.06	2.13	2.28	4.71	20466	0.94	12.94	221.9	62.72	0.94
2	N39	G	7.5	Medium	2.77	82.67	3.70	3.66	3.25	22320	2.26	17.33	236.5	60.16	0.79
3	N16	G	7.5	Medium	2.53	83.4	3.60	3.61	3.95	28217	1.82	16.6	240.9	65.26	0.78
4	N47	G	15	High	2.52	84.52	2.18	2.34	5.16	31195	0.88	15.48	182.0	59.25	0.93
5	N16	G	7.5	Medium	2.54	84.65	2.57	2.6	4.33	35281	1.19	15.35	207.4	64.06	0.95
6	N39	B	0	Burnt	2.74	80.66	3.46	2.55	3.28	19370	1.83	19.34	295.8	63.58	1.03
7	N16	B	0	Burnt	2.45	85.31	2.33	1.68	3.67	26696	1.09	14.69	311.0	71.15	1.02
8	N39	G	15	High	2.52	79.37	3.93	3.69	4.37	35396	1.75	20.63	194.8	57.44	0.81
9	N12	B	0	Burnt	2.47	84.62	3.27	3.18	4.45	26894	1.45	15.38	275.6	70.64	1.22
10	N12	G	7.5	Medium	2.26	82.74	4.02	4.19	4.87	35872	1.69	17.26	221.0	66.89	0.99
11	N12	G	15	High	2.17	81.57	3.79	3.82	4.61	49696	1.65	18.43	237.1	61.48	0.99
12	N12	B	0	Burnt	2.2	82.73	3.86	3.56	4.55	25582	1.63	17.27	274.6	70.29	1.25
13	N47	G	15	High	2.44	83.2	2.96	3.3	5.33	35237	1.17	16.8	192.9	59.92	0.8
14	N39	G	7.5	Medium	2.75	81.45	9.00	3.97	4.00	24687	1.94	18.55	234.9	64.17	0.96
15	N16	B	0	Burnt	2.53	84.98	2.66	2.19	3.56	28179	1.36	15.02	304.3	69.79	0.97
16	N47	G	7.5	Medium	2.56	87.5	1.86	1.98	4.69	22167	0.82	12.5	223.7	63.82	0.99
17	N47	G	7.5	Medium	2.56	86.33	4.21	3.12	5.08	23200	1.44	13.67	224.9	64.00	0.97
18	N47	B	0	Burnt	2.69	84.01	3.06	3.16	4.46	15907	1.39	15.99	278.8	69.08	1.05
19	N16	G	15	High	2.38	82.77	2.82	2.96	4.62	40130	1.25	17.23	249.7	59.58	0.85
20	N47	G	15	High	2.49	85.94	1.78	1.93	5.62	32496	0.66	14.06	193.2	61.96	0.99
21	N16	G	0	None	2.59	87.26	2.14	1.54	3.47	16228	1.06	12.74	315.0	71.24	0.77
22	N47	B	0	Burnt	2.69	84.39	3.45	2.98	4.46	16089	1.44	15.61	287.4	69.22	1.09
23	N16	G	0	None	2.63	85.17	2.77	2.97	3.42	16907	1.68	14.83	300.0	69.93	1.04
24	N39	G	0	None	2.69	84.01	2.86	2.8	3.35	14261	1.69	15.99	290.3	65.37	0.87

Table C-5 continued

Test	Variety	Type	% Brown leaf	Brown leaf	Brix (g/100 g)	Gravity Purity (%)	Fructose (g/100 g Brix)	Glucose (g/100 g Brix)	Cond ash (g/100 g Brix)	Colour (IU)	RS/ash	Non-sucrose (g/100 g Brix)	Cane density (kg/m^3)	Moisture %	Perc rate (m/min)
25	N16	B	0.0	Burnt	2.61	82.38	4.03	4.14	3.45	18883	2.37	17.62	297.3	69.73	0.91
26	N39	G	0.0	None	2.76	83.33	2.9	2.95	3.62	14019	1.62	16.67	294.9	63.93	0.92
27	N12	G	7.5	Medium	2.39	85.77	2.35	2.29	4.18	34021	1.11	14.23	232.5	66.38	0.85
28	N16	G	7.5	Medium	2.51	83.27	4.29	2.57	3.98	24993	1.72	16.73	225.2	65.37	0.88
29	N39	B	0.0	Burnt	2.61	78.93	4.16	2.50	3.45	23551	1.93	21.07	288.5	63.60	0.96
30	N12	G	15.0	High	2.27	79.74	3.85	4.17	5.29	44502	1.52	20.26	176.7	62.20	0.92
31	N16	G	15.0	High	2.63	80.99	3.36	3.65	4.56	42956	1.54	19.01	256.0	59.49	0.85
32	N39	G	0.0	None	2.78	82.01	3.83	3.68	3.24	15327	2.32	17.99	279.8	65.32	0.91
33	N12	G	0.0	None	2.46	86.99	2.93	3.43	3.66	24670	1.74	13.01	292.7	68.70	1.29
34	N12	G	0.0	None	2.36	86.86	3.24	3.46	3.81	21526	1.76	13.14	270.7	69.24	1.12
35	N47	B	0.0	Burnt	2.63	84.79	2.59	1.68	4.56	17161	0.94	15.21	281.3	68.68	1.15
36	N47	G	0.0	None	2.66	87.97	1.76	1.94	4.14	13860	0.89	12.03	284.0	68.11	1.07
37	N12	B	0.0	Burnt	2.32	84.05	2.92	2.56	4.31	22564	1.27	15.95	292.1	69.49	1.32
38	N12	G	15.0	High	2.25	80.89	3.63	3.93	4.89	40279	1.55	19.11	183.6	61.89	0.9
39	N39	G	15.0	High	2.64	80.68	3.38	3.15	3.79	31719	1.72	19.32	216.9	54.51	0.82
40	N12	G	0.0	None	2.38	86.97	3.04	3.07	3.78	21929	1.62	13.03	286.9	70.61	1.14
41	N47	G	0.0	None	2.76	89.86	1.46	1.40	3.99	10668	0.72	10.14	318.0	68.83	1.04
42	N16	G	15.0	High	2.39	81.17	3.49	3.73	4.60	52291	1.57	18.83	256.7	62.28	0.9
43	N39	G	15.0	High	2.61	80.84	3.59	3.62	3.83	34733	1.88	19.16	201.0	55.99	0.7
44	N39	G	7.5	Medium	2.68	82.09	3.45	2.99	3.73	26353	1.73	17.91	233.5	61.07	0.9
45	N39	B	0.0	Burnt	2.67	79.78	3.97	2.85	3.00	18304	2.28	20.22	282.9	64.41	1.05
46	N47	G	0.0	None	2.55	84.71	2.52	2.73	5.10	11531	1.03	15.29	291.8	70.44	1.04
47	N12	G	7.5	Medium	2.33	83.69	3.64	3.90	4.72	32911	1.6	16.31	223.2	64.26	1.06
48	N16	G	0.0	None	2.45	85.71	3.77	3.92	3.27	18115	2.35	14.29	307.2	69.44	0.98

Table C-6: DAC extract results for the respective experiments

Test no.	Variety	Brown leaf	Brix	Fructose	Glucose	Reducing sugars (F +G)	Gravity purity	Cond ash %	Colour	RS/ash
		%	g/100 g sample	g/100g brix	g/100 g brix	g/100 g brix	%	g/100 g brix	IU	Ratio
4	N47	15.0	4.75	0.129	0.152	0.281	81.26	4.63	29014	0.06
6	N39	0.0	6.20	0.219	0.175	0.394	80.48	3.06	20921	0.13
7	N16	0.0	5.63	0.222	0.207	0.428	83.30	3.02	20327	0.14
9	N12	0.0	5.46	0.171	0.170	0.340	82.23	3.85	24587	0.09
16	N47	7.5	6.07	0.126	0.143	0.269	84.51	3.95	19683	0.07
18	N47	0.0	5.96	0.226	0.179	0.405	81.04	4.03	17538	0.10
19	N16	15.0	4.83	0.181	0.207	0.388	79.09	4.14	32954	0.09
21	N16	0.0	5.82	0.214	0.234	0.449	82.30	3.26	15142	0.14
27	N12	7.5	5.51	0.149	0.159	0.308	82.76	3.45	31728	0.09
28	N16	7.5	5.76	0.195	0.210	0.405	81.77	3.47	25318	0.12
32	N39	0.0	6.38	0.345	0.350	0.695	79.47	2.82	16346	0.25
33	N12	0.0	5.46	0.171	0.181	0.351	85.16	3.11	17929	0.11
36	N47	0.0	5.90	0.139	0.161	0.300	86.10	3.90	28974	0.08
38	N12	15.0	4.50	0.170	0.179	0.349	79.78	4.22	43815	0.08
39	N39	15.0	5.02	0.219	0.215	0.434	77.69	3.59	30078	0.12
44	N39	7.5	5.98	0.237	0.247	0.484	79.10	3.18	31434	0.15
DAC N16	Control	0.0	5.72	0.220	0.243	0.462	83.04			
DAC N39	Control	0.0	5.92	0.231	0.265	0.496	81.93			
DAC N47	Control	0.0	5.50	0.137	0.154	0.291	85.64			

Figure C-1: Photographs of shredded sugarcane with and without brown leaf prepared in a factory shredder and in the Waddell shredder

D Scatterplots for brown leaf trials

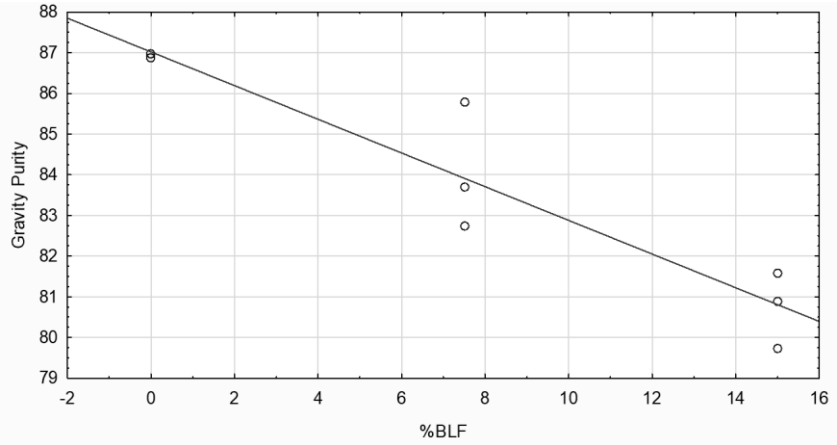

Figure D-1:Gravity purity % - N12

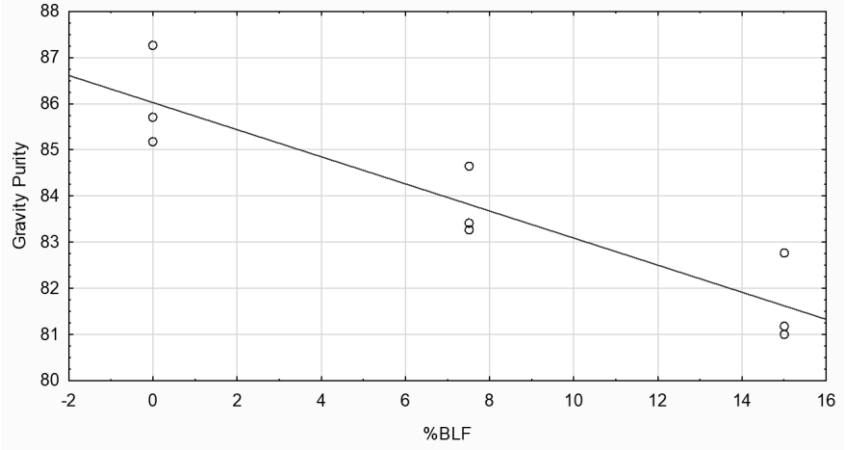

Figure D-2:Gravity purity % - N16

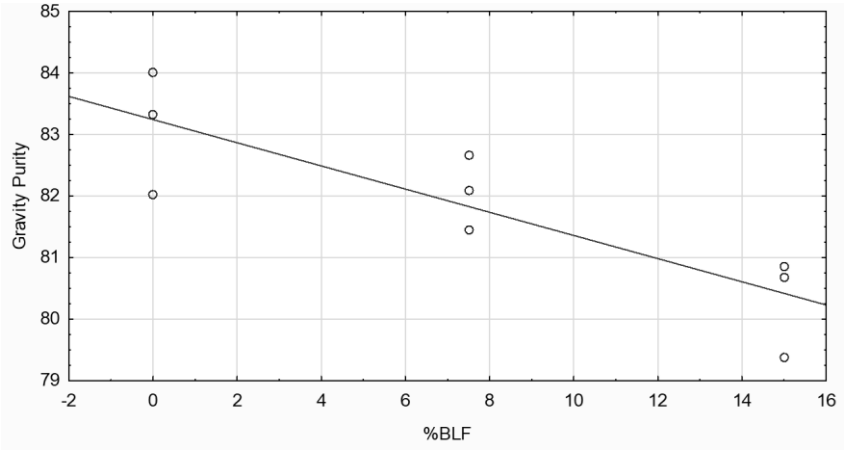

Figure D-3: Gravity purity % - N39

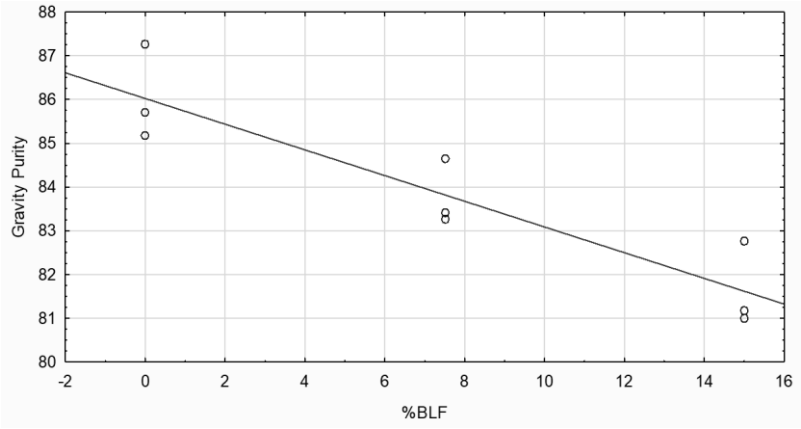

Figure D-4: Gravity purity % - N47

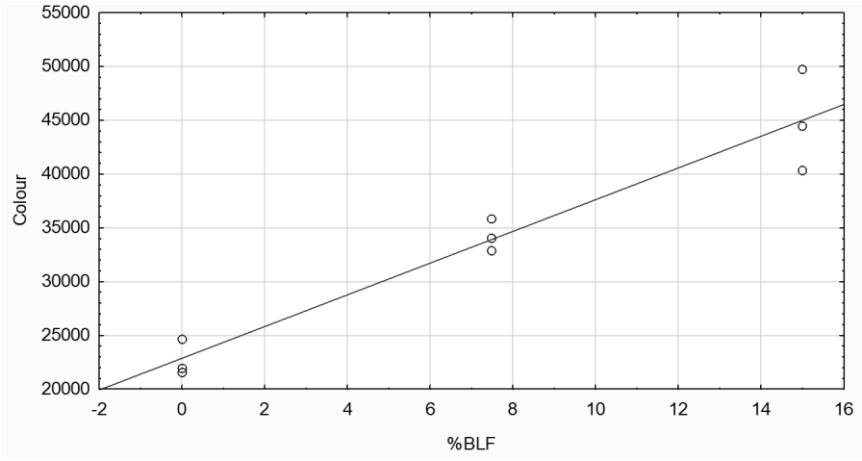

Figure D-5: Colour (ICUMSA units) - N12

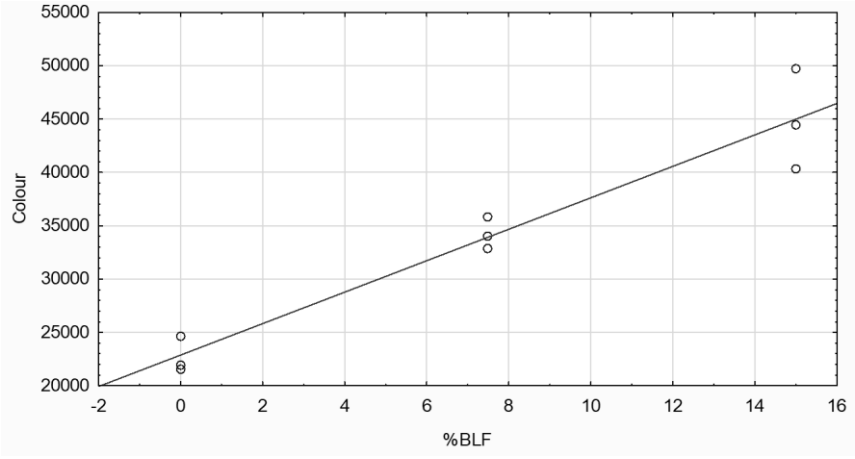

Figure D-6: Colour (ICUMSA units) - N16

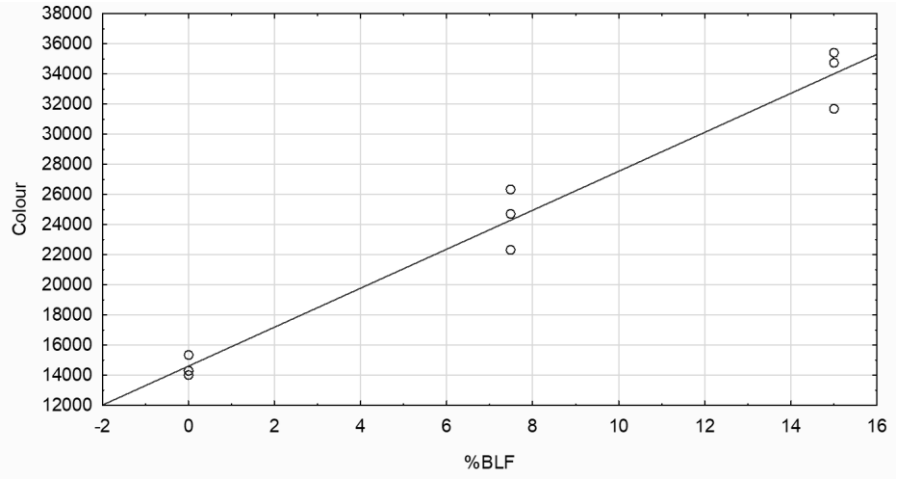

Figure D-7: Colour (ICUMSA units) - N39

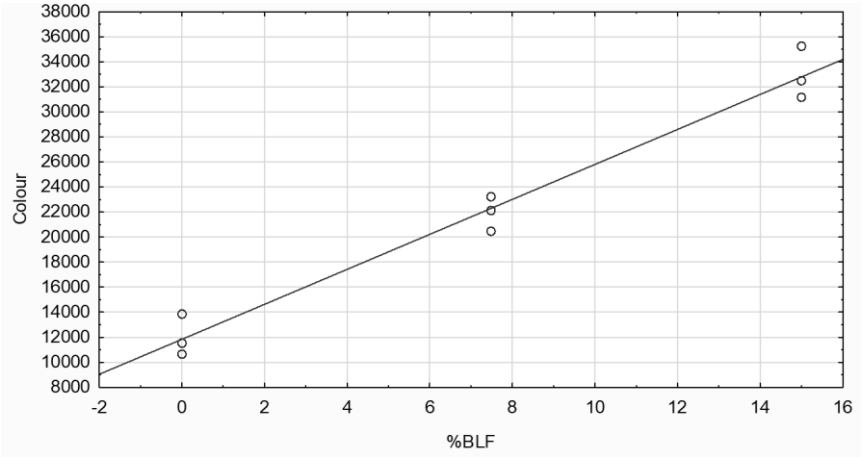

Figure D-8: Colour (ICUMSA units) - N47

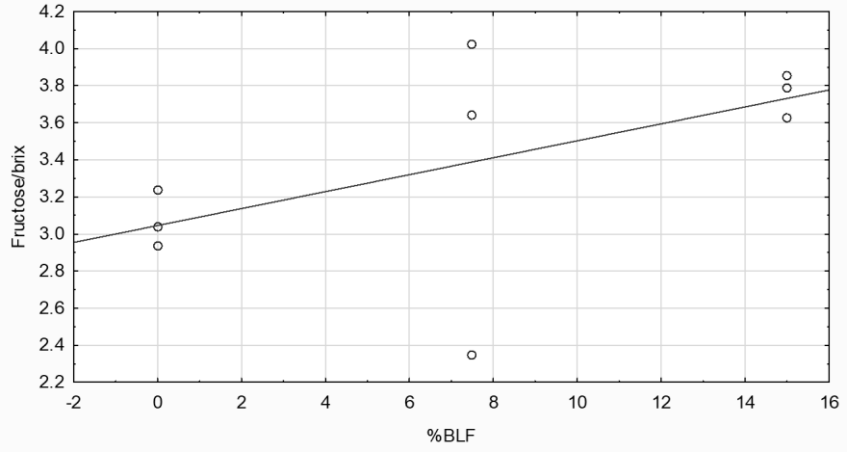

Figure D-9: Fructose/Brix (g/100 g) - N12

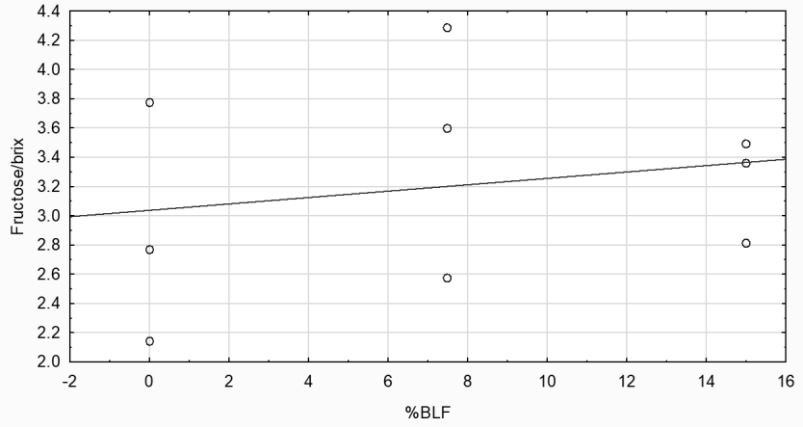

Figure D-10: Fructose/Brix (g/100 g) - N16

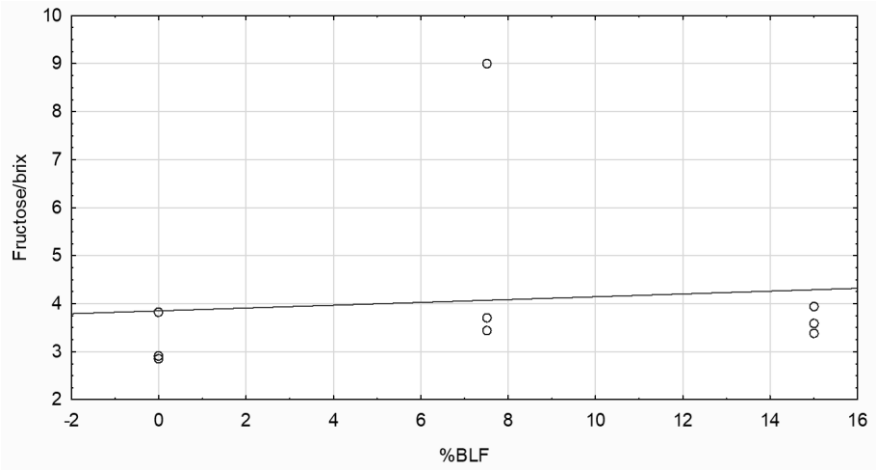

Figure D-11: Fructose/Brix (g/100 g) - N39

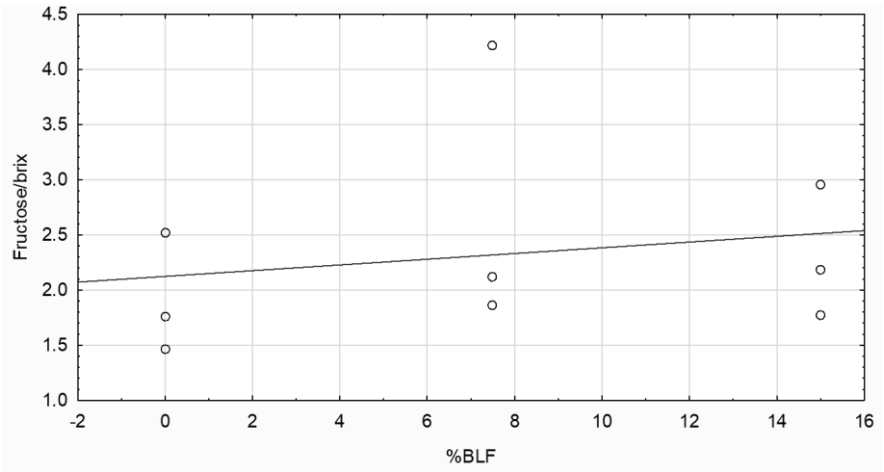

Figure D-12: Fructose/Brix (g/100 g) – N47

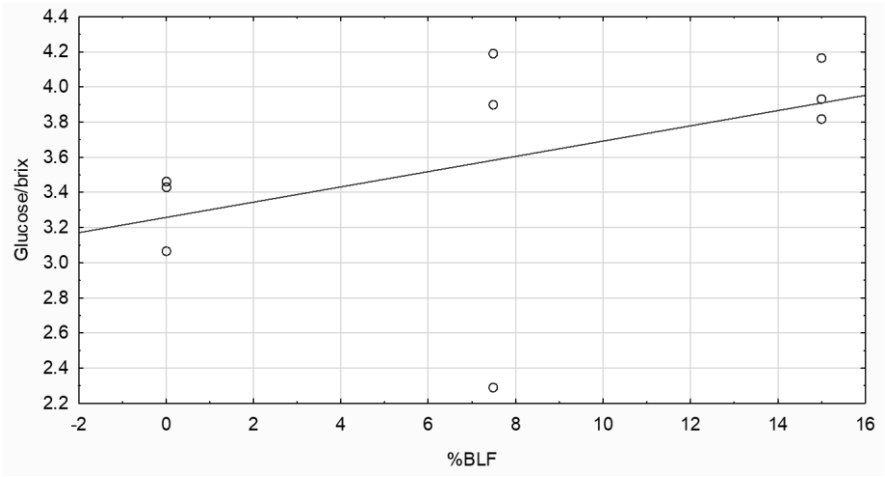

Figure D-13: Glucose/Brix (g/100 g) - N12

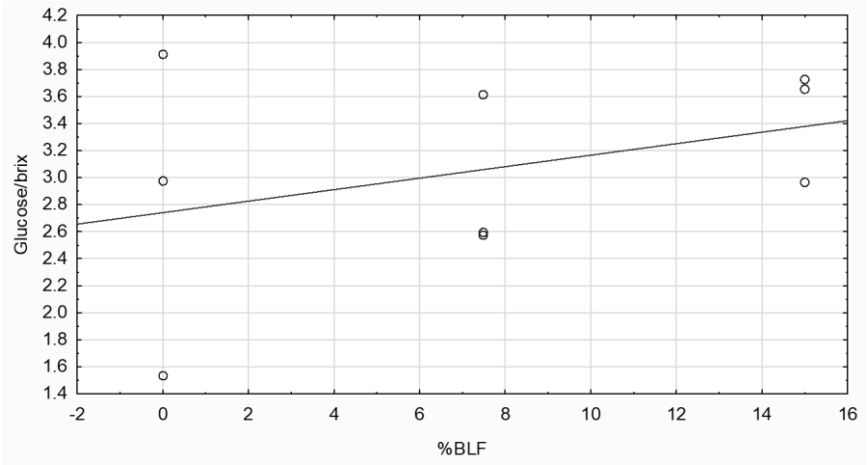

Figure D-14: Glucose/Brix (g/100 g) - N16

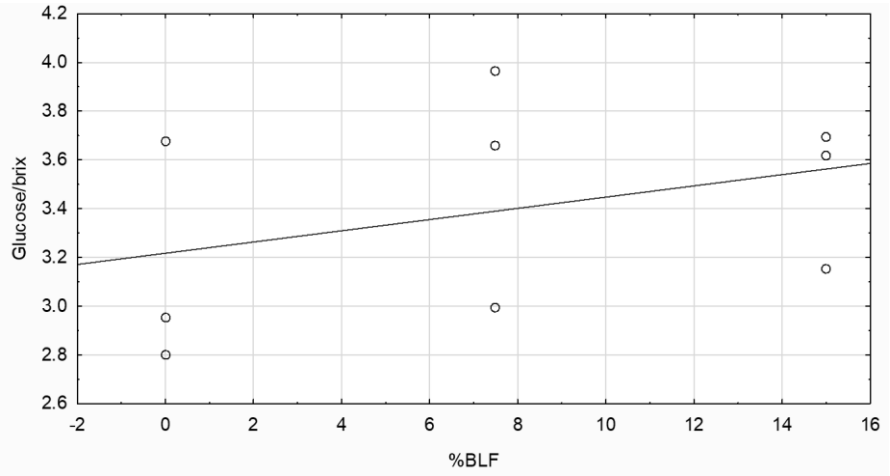

Figure D-15: Glucose/Brix (g/100 g) – N39

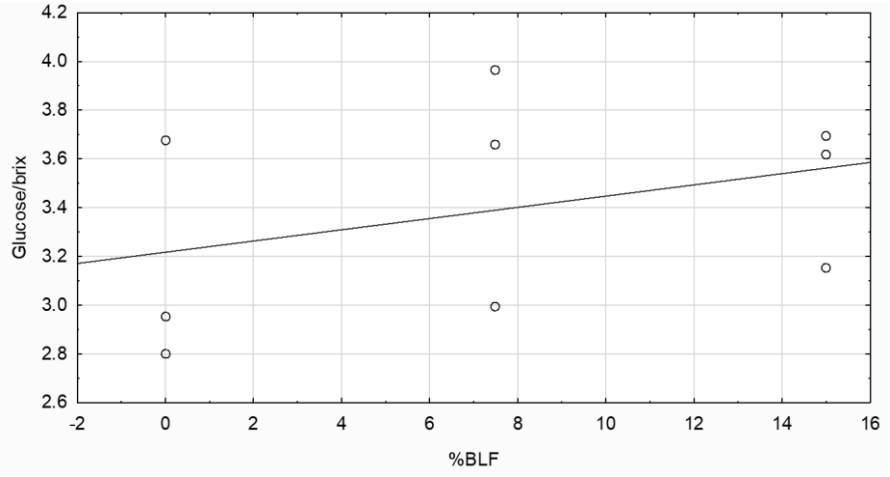

Figure D-16: Glucose/Brix (g/100 g) – N47

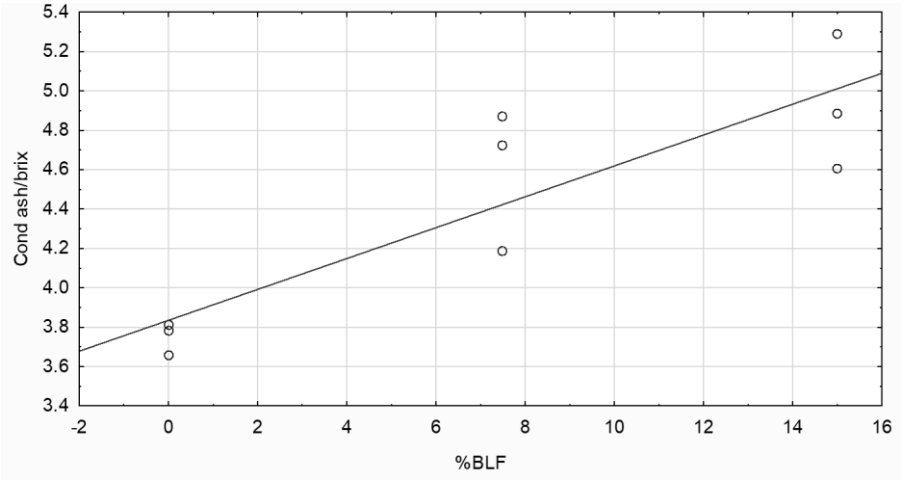

Figure D-17: Conductivity ash/Brix (g/100 g) - N12

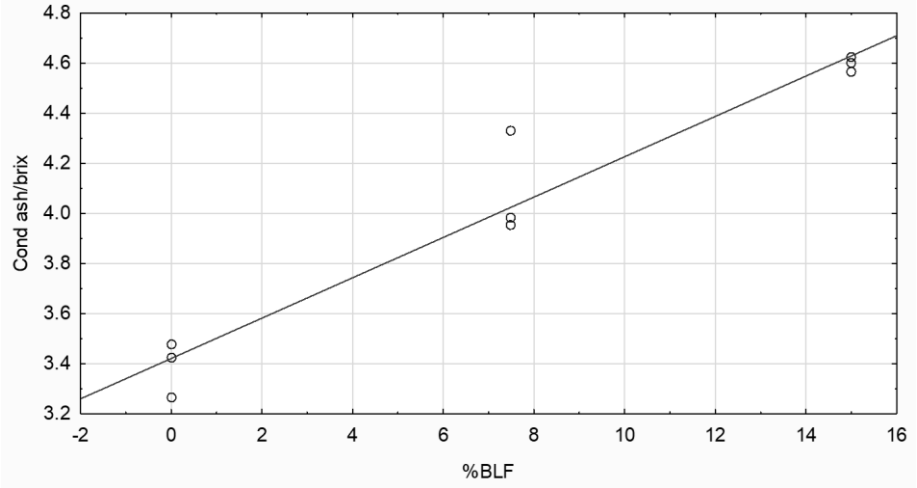

Figure D-18: Conductivity ash/Brix (g/100 g) - N16

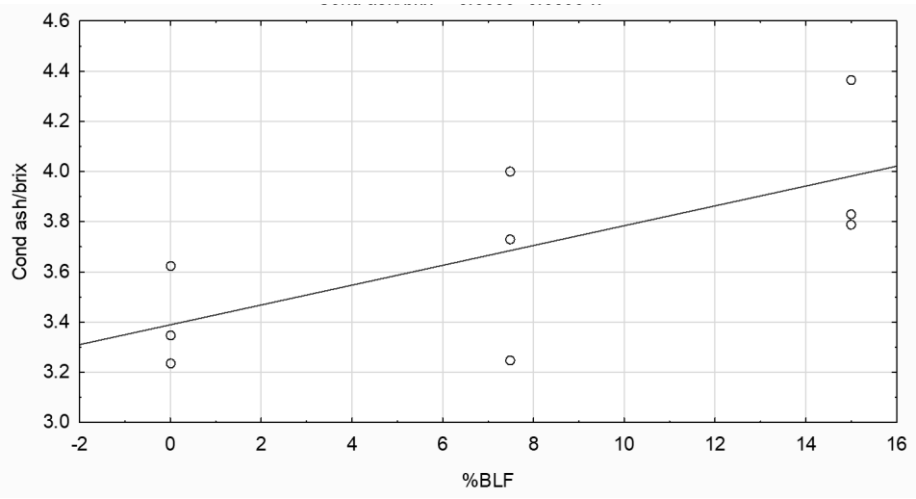

Figure D-19: Conductivity ash/Brix (g/100 g) - N39

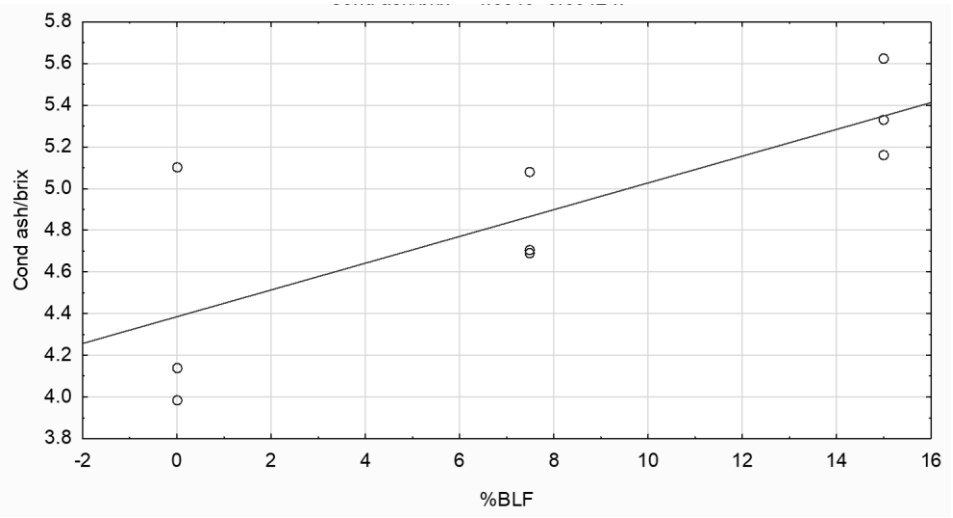

Figure D-20: Conductivity ash/Brix (g/100 g) - N47

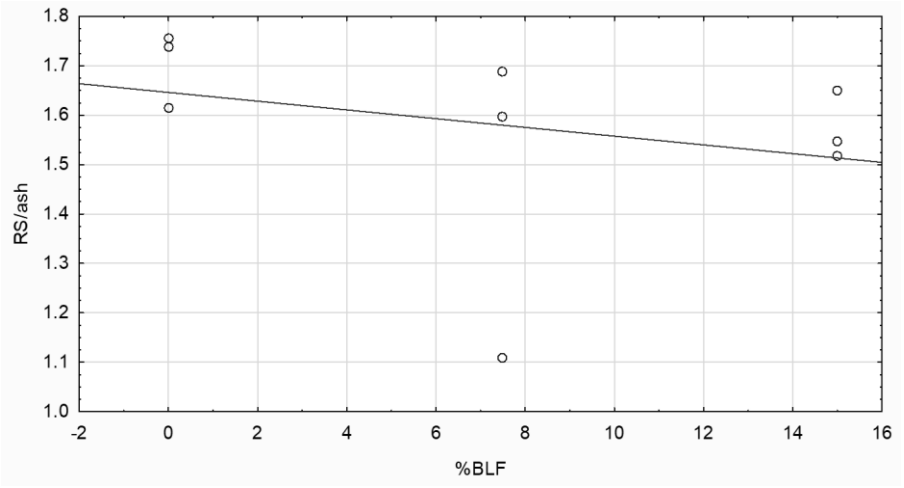

Figure D-21: Reducing sugars/ash (g/100 g) - N12

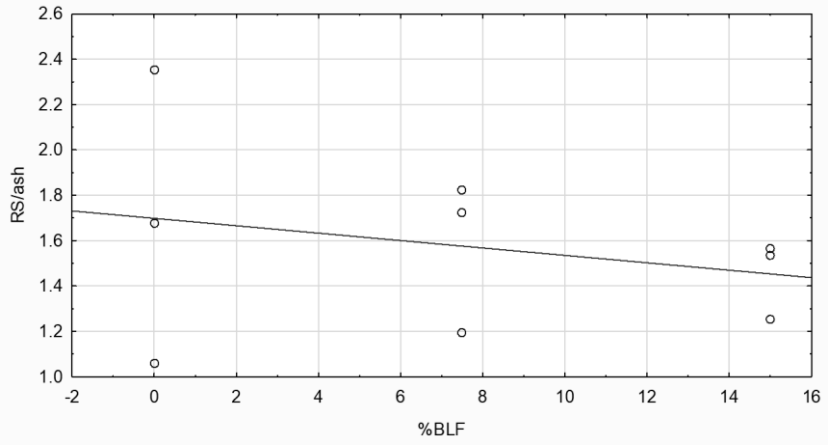

Figure D-22: Reducing sugars/ash (g/100 g) - N16

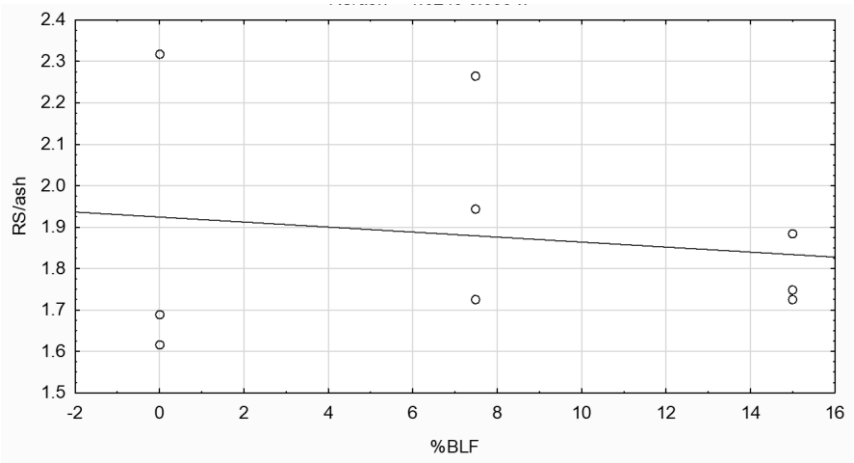

Figure D-23: Reducing sugars/ash (g/100 g) - N39

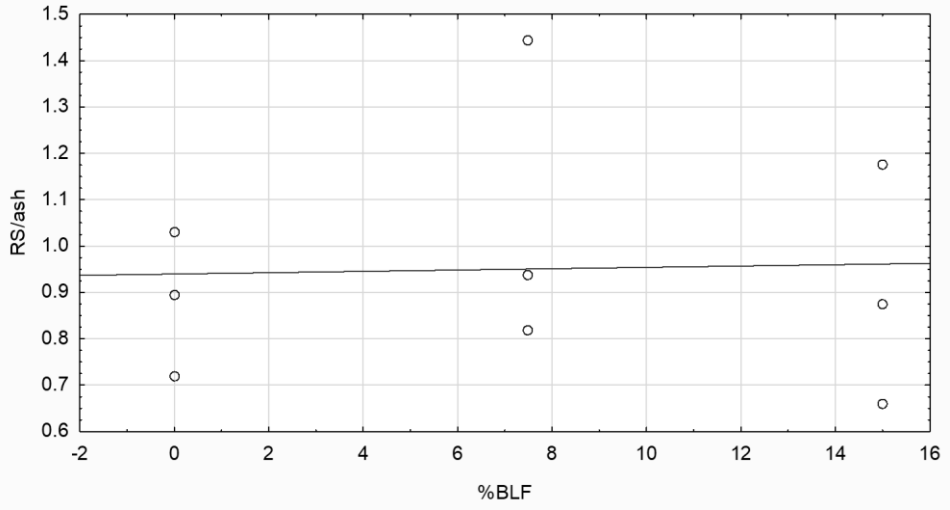

Figure D-24: Reducing sugars/ash (g/100 g) – N47

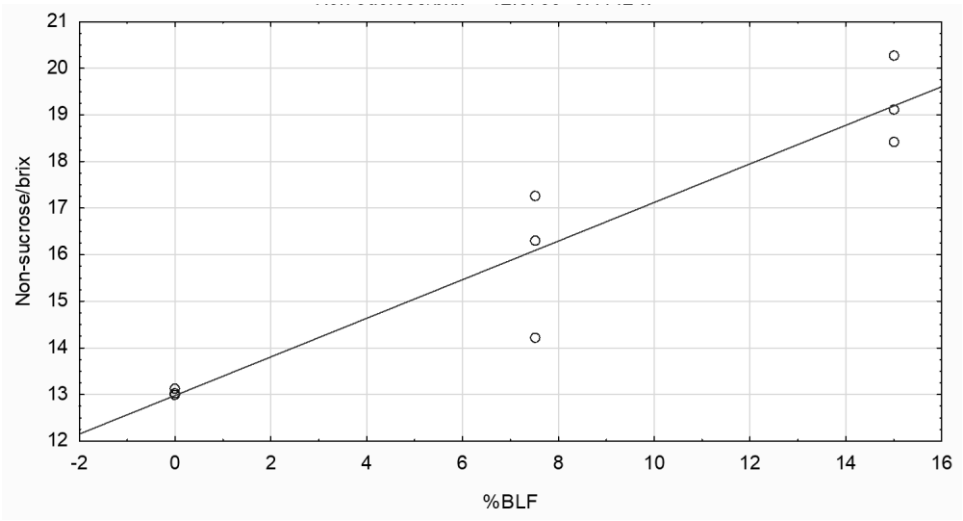

Figure D-25: Non sucrose/Brix (g/100 g) - N12

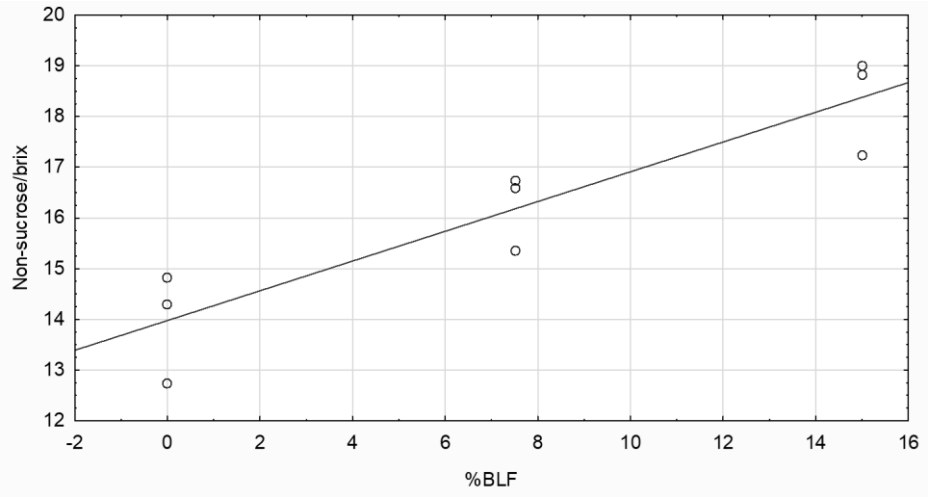

Figure D-26: Non sucrose/Brix (g/100 g) - N16

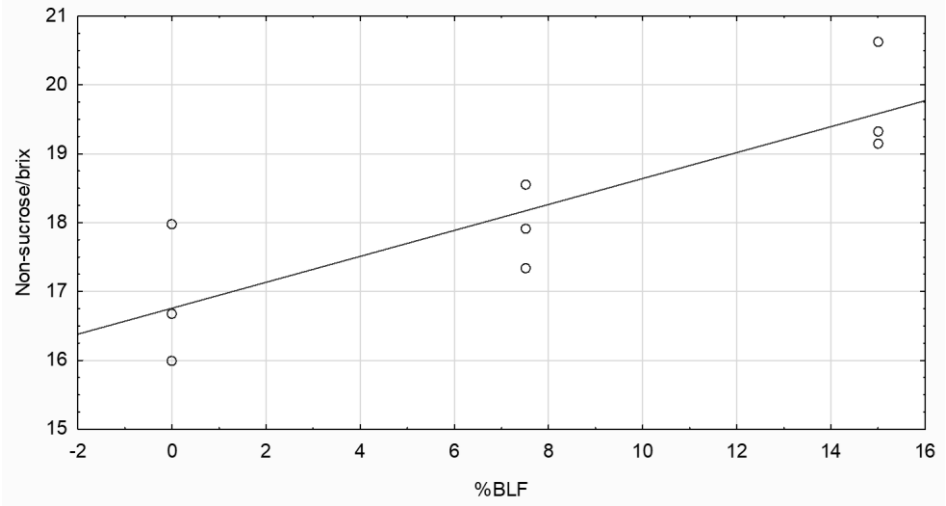

Figure D-27: Non sucrose/Brix (g/100 g) – N39

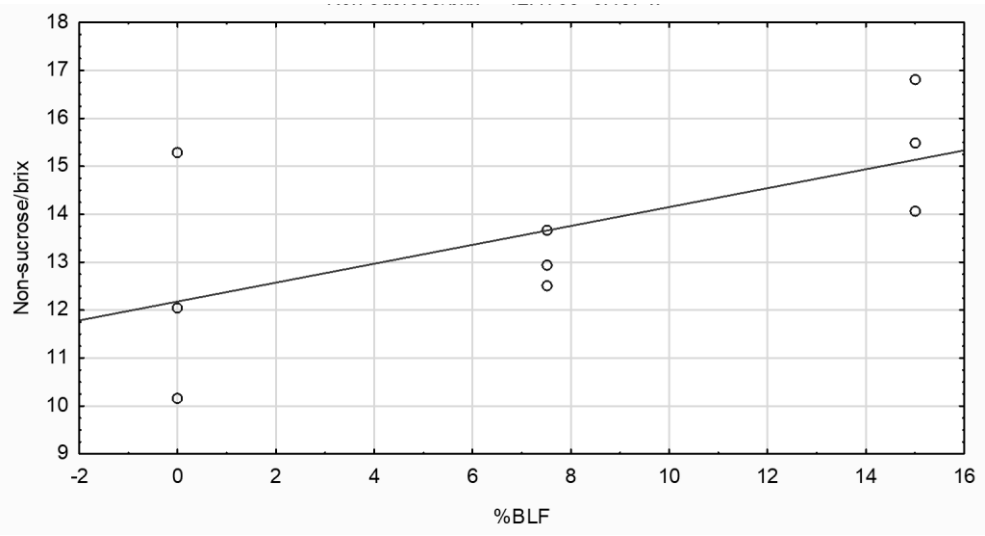

Figure D-28: Non sucrose/Brix (g/100 g) – N47

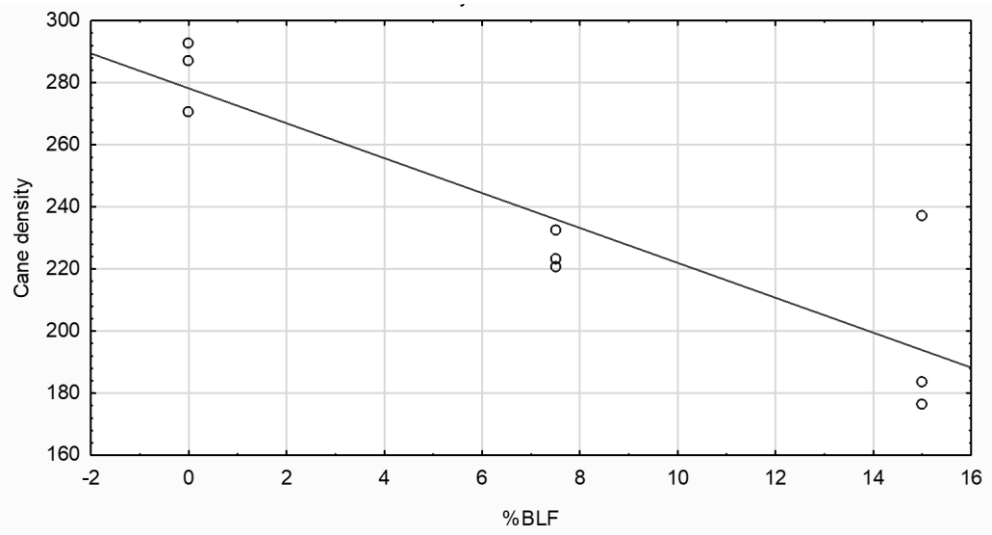

Figure D-29: Sugarcane density (g/100 g) - N12

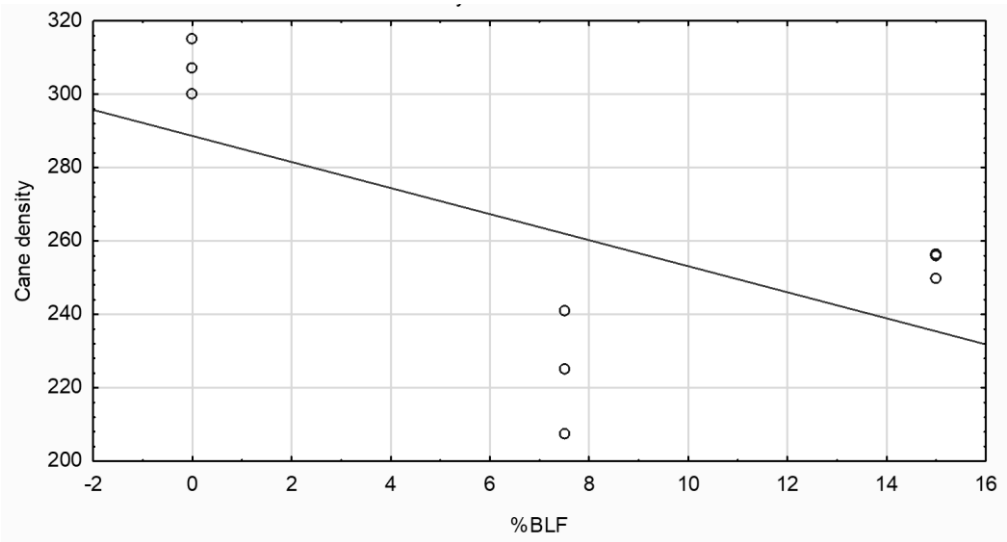

Figure D-30: Sugarcane density (g/100 g) - N16

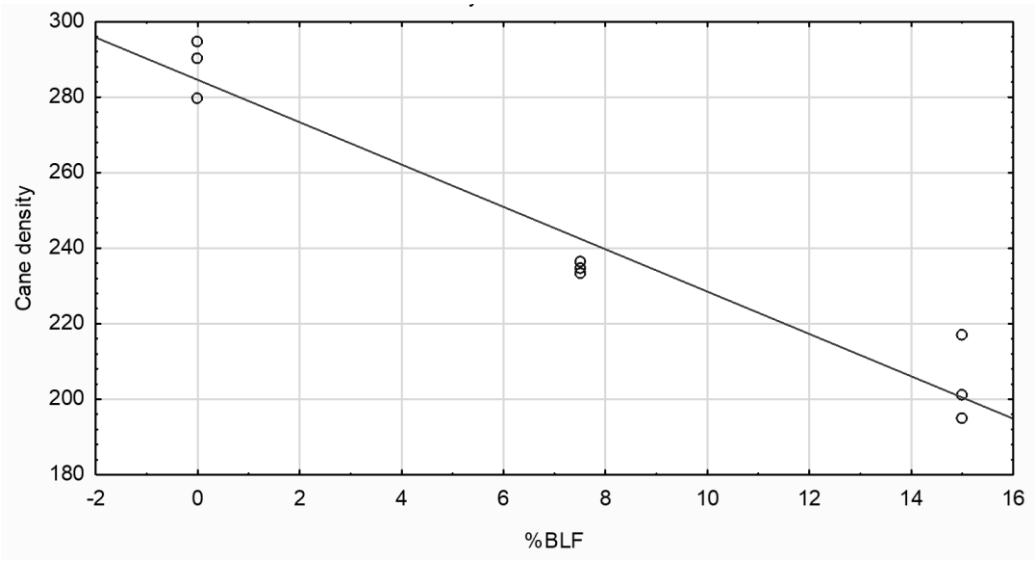

Figure D-31: Sugarcane density (g/100 g) – N39

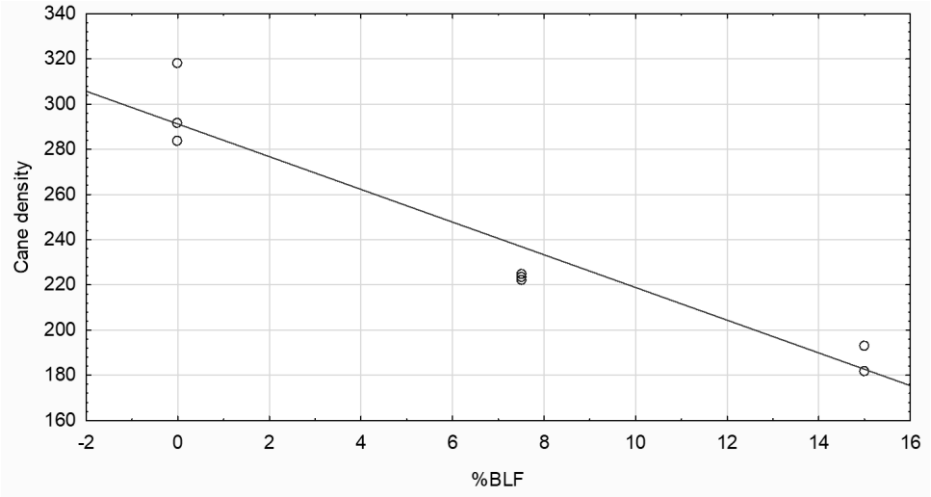

Figure D-32: Sugarcane density (g/100 g) – N47

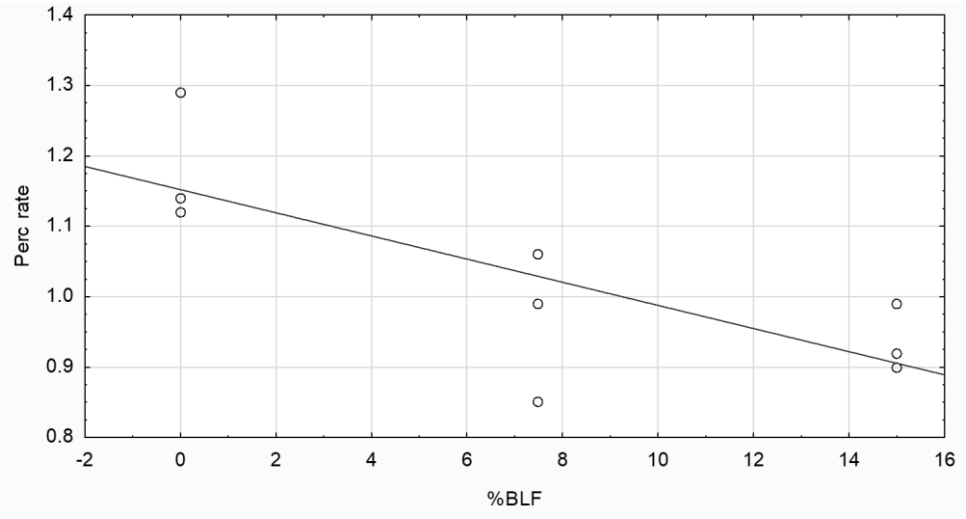

Figure D-33: Percolation rate (m/min) - N12

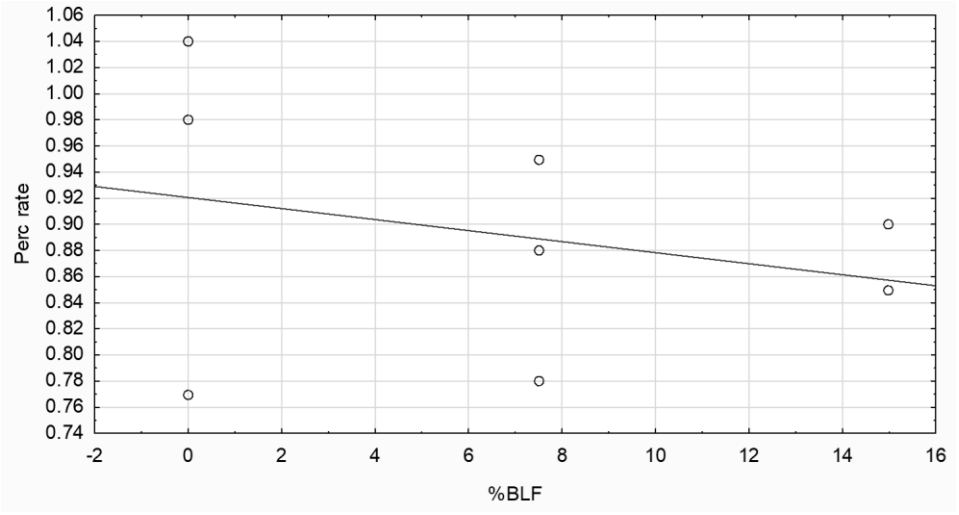

Figure D-34: Percolation rate (m/min) - N16

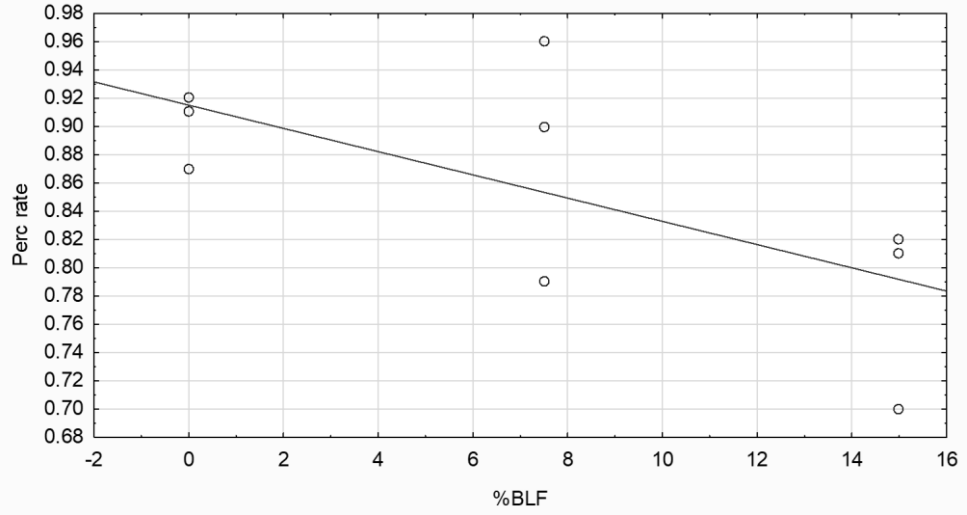

Figure D-35: Percolation rate (m/min) – N39

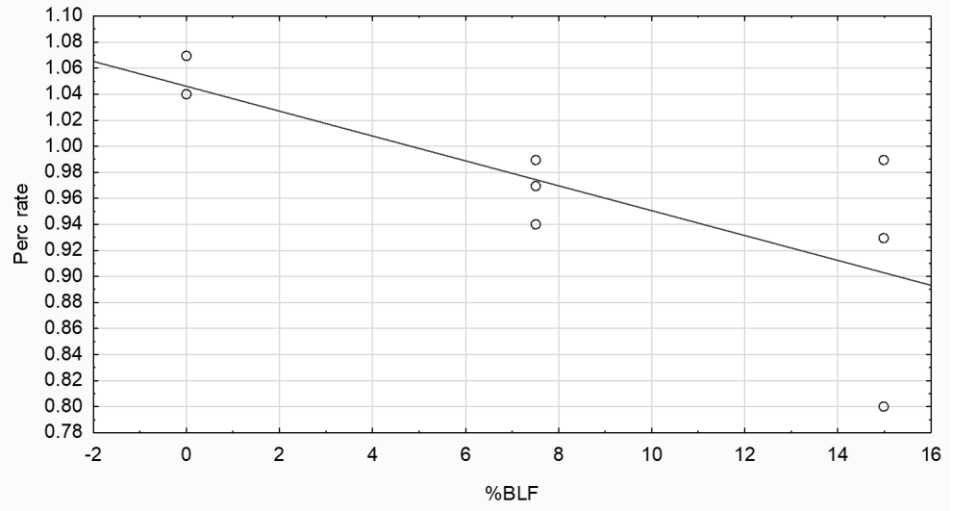

Figure D-36: Percolation rate (m/min) – N47