

# **Extraction of cellulose from cacti**

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## ABSTRACT

Paraffin is used as a main household energy source for cooking, lighting and heating by low-income communities in South Africa. It is highly inflammable and spillages from paraffin can be considered as one of the major causes of fires that lead to the destruction of dwellings in the informal settlement. The situation is made worse due to the close proximity of the dwellings to each other which cause the fires to spread very quickly from one dwelling to the next leaving suffering and most often death in its wake (Schwebel *et al.*, 2009:700). It has been shown by Muller *et al.* (2003:2018) that most of the informal rural communities use paraffin in non-ventilated and windowless environments and this causes major respiratory problems.

The government has made a huge effort towards replacing paraffin as main cooking fuel in rural and informal settlements with ethanol gel. Ethanol gel is a healthier, safer alternative to paraffin because ethanol gel does not burn unless it is contained within a cooking device that concentrates the flame. It also fails to emit lung irritants or other dangerous chemical vapours when burned indoors (Bizzo *et al.*, 2004:67).

Commercial ethanol gels are manufactured with imported gelling agents that make their costs unaffordable to the rural poor communities. It is the objective of this study to determine whether gelling agents extracted from the local endemic species of cactacea viz. *Opuntia ficus-indica* and *Cereus Jamacaru* can be used to synthesise ethanol gel comparable or better than the commercial gels. The two species chosen have been declared pests (Nel *et al.*, 2004:61) and are continuously uprooted from arable land and burned by local farmers (Van Wilgen *et al.*, 2001:162)

This study showed that *Opuntia ficus-indica* stems gave a better cellulose yield ( $15.0 \pm 6.7$  wt. %) than *Cereus Jamacaru* ( $11.5 \pm 7.8$ wt %). Chemical composition analyses and FT-IR analyses showed that the hemicelluloses and lignin were completely removed from the extracted cellulose and the extraction was more effective for *Opuntia ficus-indica* than for *Cereus Jamacaru*. Ethanol gel produced by using the extracted cellulose,

as was investigated during this study, was compared to commercial gels with respect to viscosity, burn time, calorific values and residue and a good comparison was obtained.

**Key words:** *Opuntia ficas-indica*, *Cereus Jamacaru*, ethanol gel, commercial gels, informal settlement, cellulose.

## OPSOMMING

Paraffien word hoofsaaklik gebruik as 'n huishoudelike bron van energie vir kook, beligting en verwarming in die meeste lae-inkomsteklas gemeenskappe van Suid-Afrika. Alhoewel paraffien dus baie bruikbaar is, is dit egter hoogs ontvlambaar en is die vermorsing daarvan een van die hoof oorsake van vernietigende brande in informele huisvestings. Die situasie word vererger deur die naburigheid van hierdie informele vestigings wat daartoe lei dat die brande vinniger kan versprei van een huis na 'n ander. Hierdie verwoede brande laat lyding en soms ook dood in hulle verwoestingspad na (Schwebel *et al.*, 2009:700). Volgens Muller *et al.* (2003:2018), gebruik die meeste informele plattelandse gemeenskappe paraffien in ongeventileerde en vensterlose omgewings en sodanige praktyke veroorsaak ernstige respiratoriese probleme.

Die regering het 'n spesiale poging aangewend om paraffien as hoofvorm van brandstof vir kook in plattelandse en informele gemeenskappe te vervang met etanol gel. Etanol gel is 'n gesonder en veiliger alternatief as paraffien aangesien dit nie brand nie tensy dit bevat word in 'n kookapparaat waarin die vlamme gekonsentreer word. Verder is daar aangetoon dat indien die gel binneshuis verbrand word, daar geen skadelike of gevaarlike dampe, wat tot longprobleme kan lei, afgegee word nie (Bizzo *et al.*, 2005:67).

Kommersiële etanol gel word tans vervaardig deur gebruik te maak van ingevoerde verjellings agente, wat dit egter onbekostigbaar maak vir arm plattelandse gemeenskappe. Die hoofdoelstelling van hierdie projek was om te bepaal of verjellings agente wat uit plaaslike endemiese spesies van cactacea viz.: *Opuntia ficus-indica* en *Cereus Jamacaru* ge-ekstraheer word, soortgelyke of beter etanol gel hoeveelhede en eienskappe kon sintetiseer as kommersiële verjellings agente. Die twee gekose spesies is verklaar as peste (Nel *et al.*, 2004:61) en word voortdurend ontwortel uit bewerkbare grond en deur die plaaslike boere verbrand (Van Wilgen *et al.*, 2001:162).

Vanuit hierdie studie is dit vasgestel dat *Opuntia ficas-indica* stamme 'n beter sellulose opbrengs ( $15.0 \pm 6.7$  wt %) lewer as *Cereus Jamacaru* ( $11.5 \pm 7.8$  wt %). Chemiese samestellings- en FT-IR analises het beide getoon dat alle hemisellulose en lignin heeltemal vanuit die ge-ekstraheerde sellulose verwyder was en dat die ekstraksie die effektiëste was vir die *Opuntia ficas-indica*. Etanol gel geproduseer in hierdie studie vanuit die ge-ekstraheerde sellulose is vergelyk met kommersiële gelle op basis van viskositeit, brandtyd, ontbrandingswaardes (kalorifiese waardes) en residu. 'n Goeie ooreenkoms is verkry.

**Kernwoorde:** *Opuntia ficas-indica*, *Cereus Jamacaru*, etanol gel, kommersiële gel, informele huisvestings, sellulose.

## **DECLARATION**

I, Moses Seleke Monye, hereby declare to be the sole author of the dissertation entitled:

### **Extraction of cellulose from cacti**

Submitted in partial fulfilment of the requirements for the degree of Master of Science in  
Engineering Sciences in the Faculty of Engineering of the North-West University,  
Potchefstroom Campus

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Moses Seleke Monye  
Potchefstroom

## DEDICATION

*I declare by The Power of the Blood of Jesus that the ideas presented in this dissertation shall prevail above all adversity and shall be the light to mankind.*

This dissertation is dedicated to my son ITHUTENG and my late Father, MR. JACOB MOABI MONYE. His caring attitude, principles, discipline and simplicity, inculcated in my mind will be cherished in all walks of my life. Special thanks to my mother, Elizabeth Lefitile Monye and my pillars of strength: Joseph, Harry and Solomon Monye, Mighty and Zips Kole, Ndaba and Lucy Mokwayi, Thabo and Rose Khwinana, Tabea Monye, Shirley and Lerato Molale.

*"The world is a dangerous place not because of those who do evil but because of those who look and do nothing" Albert Einstein*

*"Never walk on the travelled path, because it only leads you where the others have been." Grahan Bell*

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*“Gratitude unlocks the fullness of life. It makes sense of our past, brings peace for today, and creates a vision for tomorrow”. ~Melody Beattie*

*“What we do for ourselves is mortal, for it dies with us, what we do for others and the world is immortal for it remains with them in our absence” Albert Pike (1809-1891).*

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**ABBREVIATION**

<b>Acronyms</b>	<b>Definition</b>
ADF	Acid detergent fibre
ADL	Acid detergent lignin
AGUs	D-anhydroglucopyranose units
C <sub>2</sub> H <sub>2</sub> OH	Ethanol
C <sub>6</sub> H <sub>12</sub> O <sub>6</sub>	Glucose
CF	Crude fibre
DM	Dry matter
DME	Department of Minerals and Energy
ERC	Energy Research Centre
FAO	Food and Agricultural Organization
FTIR	Fourier transform infrared spectroscopy
GFS	Gel Fuel Stove
GHG	Greenhouse gasses
GigaWatt (GW)	1GW = 1000MW
HHV	Higher Heating Value
IDF	Insoluble dietary fibre
IEA	International Energy Agency
IEP	Integrated Energy Plan
IFPRI	The International Food Policy Research Institute
IPCC	Intergovernmental Panel on Climate Change
LHV	Lower Heating Value
MDGs	Millennium Development Goals
MJ	Mega Joule

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**ABBREVIATIONS**

<b>Acronyms</b>	<b>Definition</b>
MW	Megawatt (1,000,000 W)
NDF	Neutral detergent fibre
NGOs	non-government organizations
NIST	National Institute of Standards and Technology
NREL	National Renewable Energy Laboratory
OECD	Organization for Economic Cooperation and Development
OM	Organic matter
PSASA	Paraffin Safety Association of Southern Africa
RET	Renewable Energy Technology.
SABS	South African Bureau of Standards
SANS	South African National Standards
SAPIA	South African Petroleum Industry Association
SDF	The amount of soluble dietary fibre
SEM	Scanning electron microscope
STDEV	Standard Deviation
TDF	Total dietary fibre
UN	United Nations
USA	United States of America
USDA	United states Department of Agriculture
WBC	Water binding capacity
WUE	Water-use efficiency

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## TERMINOLOGY

Term	Meaning
Bio-energy	Energy derived from biological source
Bio-ethanol	Fuel derived from the fermentation of sugars and starch
Bio-fuels	Fuel produced directly or indirectly from biomass. This includes bio-ethanol (ethanol or ethyl alcohol), biodiesel, biogas, gel fuels and biomass gases
Biogas	Fuel or combustible gas derived from the microbial digestion of Human and animal excreta or organic wastes.
Biomass	Plant material or animal wastes used as a source of fuel or other industrial Products
Energy crop	Woody or herbaceous crop grown specifically for its fuel value
Gel fuels	Bio-ethanol-based fuel which has been solidified using gelling agents
Greenhouse Gases (GHGs)	Gases primarily carbon dioxide, methane and nitrous oxide in the earth's lower atmosphere, that trap heat, thus causing an increase in the earth's temperature and leading towards the phenomenon of global warming.
Hemicellulose	Heterogeneous group of branched polysaccharides
Lignin	Complex phenolic polymers that fills spaces in the cell wall between cellulose, hemicelluloses and pectins. It confers mechanical strength to the cell wall and is a major component of secondary cell wall of trees.
Mercerization	The process involving swelling of native cellulose I fibres in concentrated sodium hydroxide, followed by formation of cellulose II upon removal of the swelling agent (O'Sullivan, 1997:185).
Renewable energy	Energy produced and/or derived from sources infinitely renovated (hydro, solar, wind) or generated by combustible renewable biomass
Renewable energy sources	Sun, wind, biomass, water (hydro), waves, tides, ocean current, geothermal, and any other natural phenomena which are cyclical and non-depletable.
Renewable-technology	The technology that converts a primary renewable source of energy or energy resource to the desired form of energy service.

## SYMBOLS

Symbols	Description	Unit
$P_0$	Radiant power of incident light to sample solution	
$\epsilon$	Molar absorbtivity	$\text{L mol}^{-1} \text{cm}^{-1}$
$n$	Number of samples	
$W$	Weight of starting material	g
$XA$	Concentration of component A	$\text{g.g-1}$
$XB$	Concentration of component B	$\text{g.g-1}$
$Y$	Yield	$\text{g.g-1}$
$\sigma$	Standard deviation	
$A$	Absorbance	
$P$	Radiant power of light leaving the sample solution	
$T$	Transmittance	
$b$	Path length	cm
$c$	Concentration of compound in solution	$\text{mol L}^{-1}$
$m_{cellulose}$	Mass of cellulose from final step of extraction	
$m_{pulp\ of\ cacti}$	Pulp of cacti used as starting sample for extraction	
$\bar{x}$	The mean of the data set	

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## CHAPTER 1 - GENERAL INTRODUCTION

*"A weed is a plant whose virtues have not been discovered"*

Ralph Waldo Emerson

### OVERVIEW

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This chapter gives a brief overview of the study. The background and motivation for the investigation is given in Section 1.1. The aims and objectives of the study are described in Section 1.2. Section 1.3 provides the scope of the dissertation and investigation.

---

#### 1.1 Background and Motivation

##### 1.1.1 Energy sources in informal settlements

Energy plays a pivotal role in the development and the creation of wealth (Balat 2006:517). South Africa's current energy sources are fossil fuels (coal, petroleum based fuels such as gasoline, diesel fuel, paraffin and gas (Department of Mineral and Energy, 1998:11). The use of fossil fuels has brought about serious environmental problems that include among others, air pollution and climate change (Winkler, 2005:27).

It is a challenge for South Africa to provide electricity to all the people and as a result most communities in informal settlements rely on wood, gas and paraffin for heating cooking and lighting.

##### 1.1.2 Effects of paraffin

Paraffin is a mixture of hydrocarbon similar to jet fuel in chemical composition (Bizzo *et al.*, 2004:61) and releases toxic fumes when it burns (Schwebel *et al.*, 2009:700). In poorly designed appliances, paraffin can ignite at a rate sufficient to raise the temperature to over 400°C within 30 seconds. In this way many shacks are torched. Paraffin is highly inflammable and spillages from paraffin can be considered one of the major causes of fires that lead to the destruction of dwellings in informal settlements. The situation is made worse, due to the close proximity of the dwellings to each other

which cause the fires to spread very quickly from one dwelling to the next leaving suffering and most often death in their wake (Schwebel *et al.*, 2009:700).

It has been shown that most of the informal rural communities use paraffin in non-ventilated and windowless environments and this causes major respiratory problems (Muller *et al.*, 2003:2018). Paraffin has the same colour and appearance as water and in some places is stored in re-used beverage containers without child-resistant caps (Schwebel *et al.*, 2009:700). Unsupervised children are at high risk of consuming paraffin. In a study conducted in a rural South African hospital, unintentional ingestion of paraffin occurred more amongst children younger than 5 years with boys leading the list (Malangu *et al.*, 2005:55). In most cases ingestion of paraffin leads to death. Kulati (2011:4) reported that paraffin is the leading cause of unintentional death in informal settlements, accounting for about 54% of total deaths.

The health hazards caused by paraffin place a huge burden on impoverished families and great pressure on the health system. To improve the lives of the rural poor communities the United Nations (UN) set targets for governments to implement renewable energy technologies (RETs) (Annan, 2005:56).

As part of the move towards renewable and cleaner energy the South African government has been engaged in huge efforts towards replacing paraffin for ethanol gel as a main cooking fuel in rural and informal settlements (Biofuels Industrial Strategy of the Republic of South Africa, 2007: 4).

## **1.2 Aims and Objectives**

The main aim and objectives of this study include the following:

- To extract gelling agents from the two local cacti namely *Opuntia ficus-indica* and *Cereus Jamacaru*.
- To synthesise different ethanol gels by using cellulose components extracted from *Opuntia ficus-indica* and *Cereus Jamacaru* cacti.
- To evaluate the synthesised ethanol gels.
- To compare synthesised ethanol gels' performances with those of commercial available gels.

### **1.3 Scope of the Investigation**

In order to fulfil the aims and objectives as given in Section 1.2, the following is required from the various sections of the dissertation.

#### **Chapter 2 - Literature Review**

- A literature study on the classification of the South African cacti.
- The study of invasive species in South Africa.
- The study of the morphology of *Opuntia ficus-indica* and *Cereus Jamacaru*.
- A literature study on cellulose and plant cell walls.
- Study on the gelation process.

#### **Chapter 3 - Experimental**

- Planning and description of the experimental set-up.
- Description of reagent used.
- Characterisation of cellulose.
- Synthesis of ethanol Gel.
- Characterisation of ethanol gel.
- Comparison of ethanol gel with commercial gels.

#### **Chapter 4 - Results and Discussion**

- Extraction results of cellulose from *Opuntia ficus-indica* and *Cereus Jamacaru*.
- Results from characterisation of cellulose.
- Results from the characterisation of ethanol gel.

#### **Chapter 5 - Conclusion and Recommendation**

- Concluding remarks based on the results obtained, are presented.
- Recommendations for future research are presented.

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## CHAPTER 2 - LITERATURE REVIEW

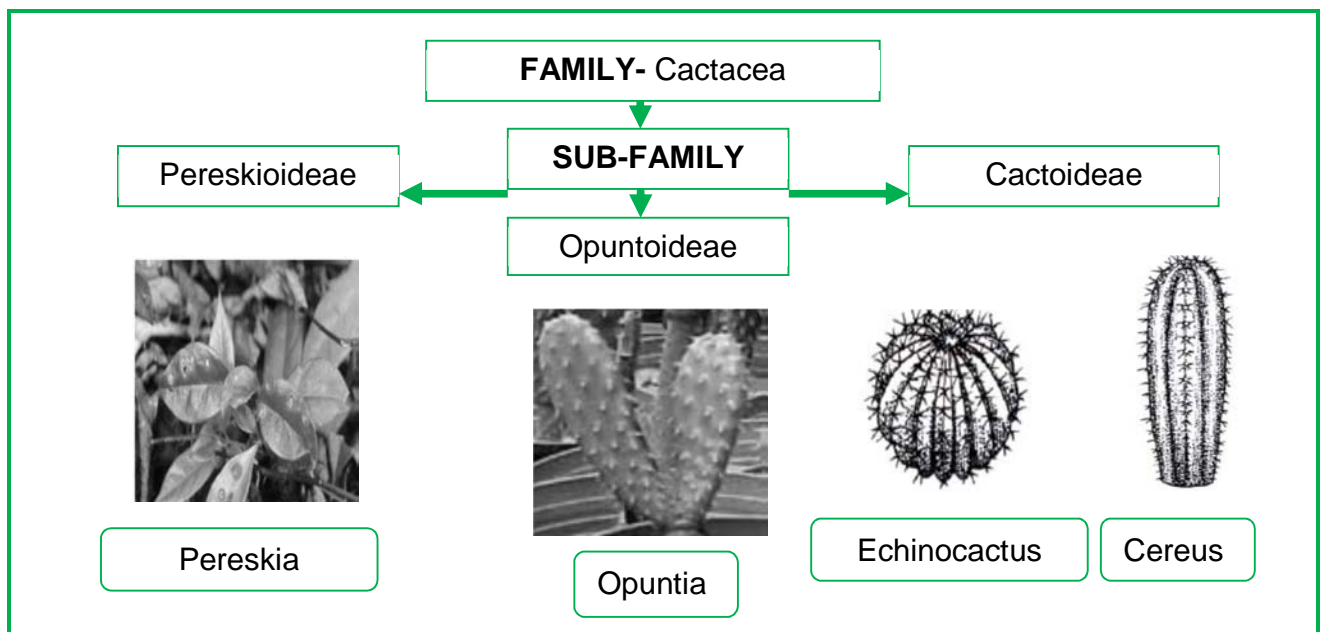
*“There is one thing stronger than all the armies of the world and that is an idea whose time has come” Victor Hugo*

### OVERVIEW

The objective of this study is to extract cellulose from two species of cacti which are *Opuntia ficas-indica* and *Cereus Jamacaru*. The cellulose is then used to synthesise ethanol gel. This chapter is divided into five sections: Section 2.1 Classification of Cacti. Section 2.2 *Opuntia ficas-indica*. Section 2.3 *Cereus Jamacaru*. Section 2.4 Polymer components of the plant cell wall and Section 2.5 Ethanol gel.

#### 2.1 Classification of cacti.

The Cactacea are dicotyledonous perennial plants (Karimi *et al.*, 2010:31) of diverse morphology that originated from North America and Brazil (Rebman & Pinkava, 2001:474). The phylogeny of the Cactacea is illustrated in Figure 2.1



**Figure 2.1** Classification of cacti (Kirkpatrick *et al.*, 2009:3)

The cacti are classified into three subfamilies: the Pereskioideae, the Opuntioideae and the Cactoideae (Griffith & Porter, 2009: 107; Rebman & Pinkava, 2001: 475; Kirkpatrick *et al.*, 2009: 1). The cacti subfamilies share the ability to store water and as such are known as succulent plants (Oldfield, 1997:1).

### **2.1.1 Invasive weeds**

According to the National Environmental Management: Biodiversity Act, No. 10 of 2004, “invasive species” means, any species whose establishment and spread are outside of its natural distribution range, threaten ecosystems, habitats or other species and has potential of causing serious harm to the environment. South Africa, like all countries worldwide finds itself on the receiving end of invasive weeds and plants (Richardson & Van Wilgen, 2004:43 ;Henderson, 2009:3).

When a natural ecosystem is invaded by alien plants, it loses its potential use. The loss of the use of a habitat has cost implications to the country. According to Van Wilgen *et al.* (2001:146) clearing alien plant invasions had cost the South African government around US\$ 1.2 billion. When opportunity costs of using the ecosystem based on its potential are added, the costs can be staggering, thus the economic burden on governments cannot be underestimated (De Lange & Van Wilgen, 2010:4113). Instead of eradicating invasive species through pesticide or biological agents (Zimmermann *et al.*, 2001:543) some creative ways of utilising invasive species need to be found.

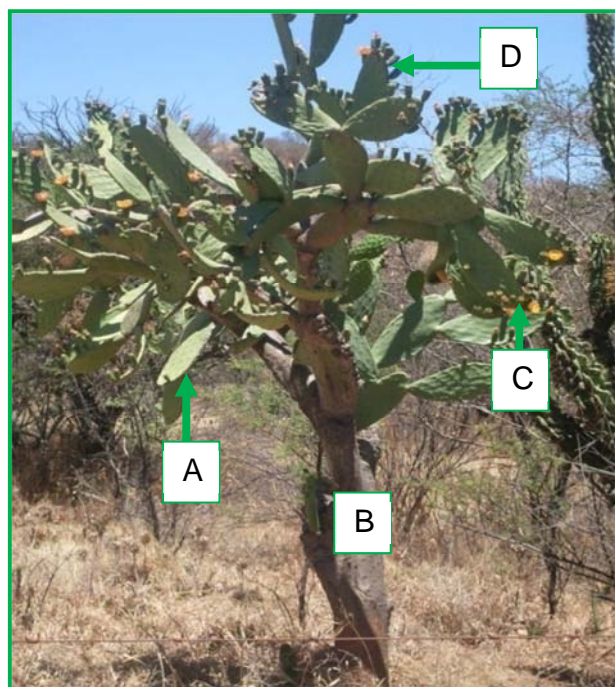
### **2.2 *Opuntia ficas-indica***

*Opuntia ficas-indica* belongs to the sub-family Opuntioideae (illustrated in Figure 2.1) and is known to have 220 to 350 species (Griffith & Porter, 2009:107). *Opuntia ficas-indica* originates from North America (Piga, 2004:9) where it is cultivated for fruit production (Pimienta-Barrios, *et al.*, 2000:74), fodder for livestock and food, known as “Nopalitos” (Saenz, 1996:89). *Opuntia ficas-indica* species are spread throughout many parts of the world such as Australia, Africa (Potgieter, 2007:7) and the Mediterranean (Piga, 2004:9). One of the main factors for the spread of *Opuntia ficas-indica* in the last 500 years was the association of *Dactylopius coccus* (an insect that produces cochineal

as dye agent) (Cha´vez-Moreno, *et al.*, 2009). Countries that wanted to produce carmine dye for food and cosmetic industry planted *Opuntia ficas-indica* as such contributed to the spread. (Cha´vez-Moreno, *et al.*, 2009). *Opuntia ficas-indica* and *Cereus Jamacaru* were introduced in South Africa through innocent human activities (i.e. as garden ornaments) or for agricultural benefit (i.e. prevention of soil erosion) (Henderson, 2009:4). According to Moran, *et al.* (1976:281) *Opuntia ficas-indica* was first recorded in 1858 by McGibbon.

### 2.2.1 Morphology

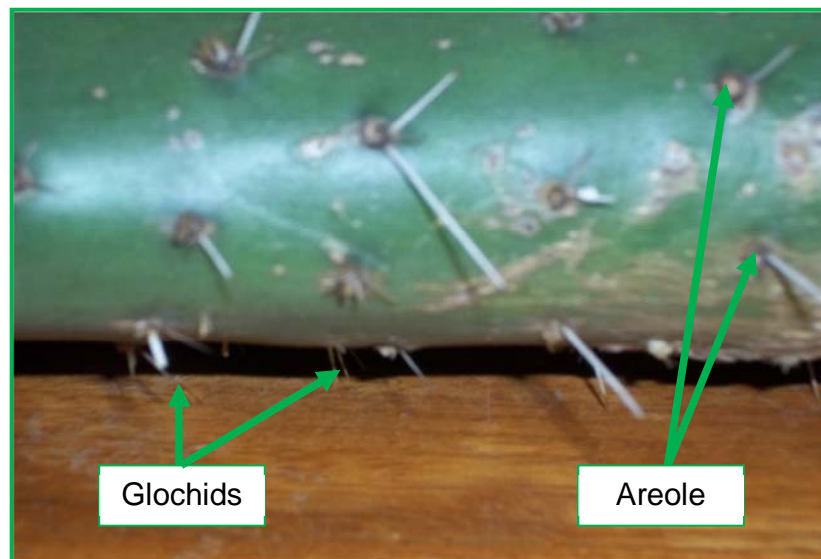
As a succulent plant *Opuntia ficas-indica* has specialised leaves (Cladodes) used to store water and function as a photosynthetic organ during times of drought (Ogburn and Edwards. 2010:183.). The cladodes can take different forms, e.g. flat cylindrical, oval or globose depending on the cultivar. *Opuntia* plants can grow to a size of up to 2m (Altesor & Ezcurra, 2003:557). The *Opuntia ficas-indica* plant as it appears at the Vredefort Dome (“Koepel”) is shown in Figure 2.2.



**Figure 2.2** *Opuntia ficas-indica* plant in the Vredefort Dome

[A. Leaves    B. Stem    C. Flowers    D. Fruit]

*Opuntia ficas-indica* bears the distinguishing feature of all cacti, namely the felted short-shoot, termed the areoles, and from these spines develop. The spines developing from the areole are of two kinds, i.e the permanent and the easily detachable ones known as glochids (Kirkpatrick *et al.*, 2009:4). The permanent spines are hard and spiky. The glochids are like barbed hair and break off easily piercing the skin and are very difficult to remove (Rebman & Pinkava, 2001:476). The glochids and areoles are illustrated in Figure 2.3 and the fruit of *Opuntia ficas-indica* is presented in Figure 2.4.



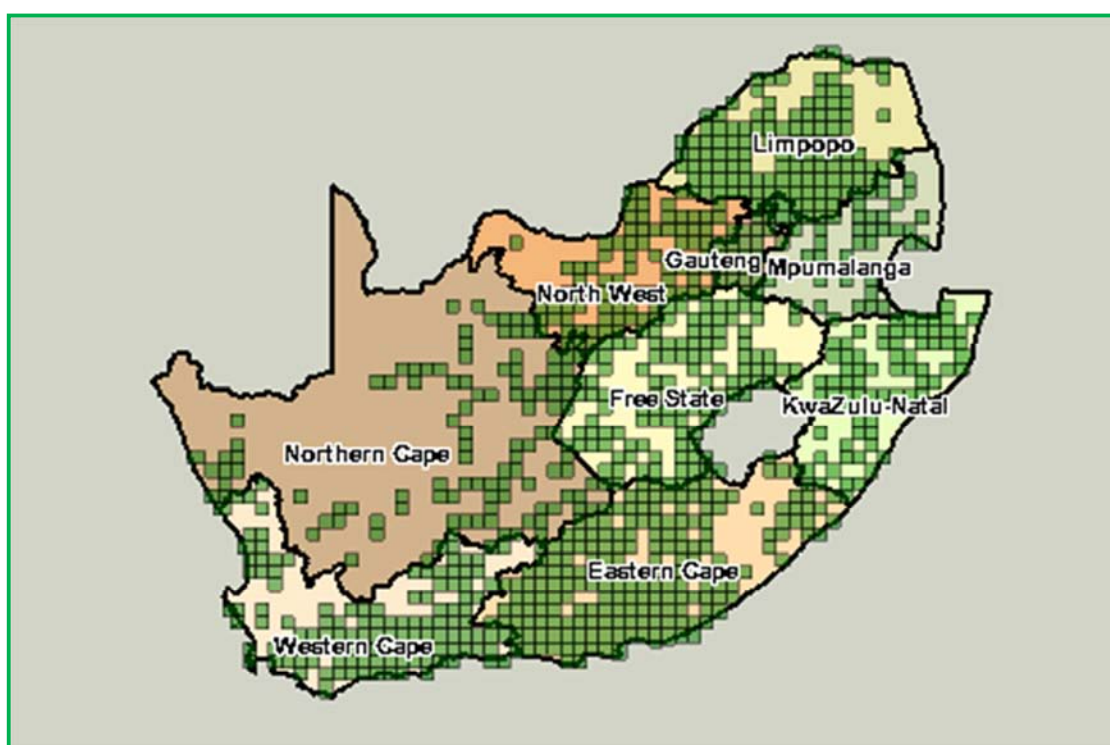
**Figure 2.3** Areole with spines and tuft of glochids



**Figure 2.4** Fruit of *Opuntia ficas-indica*

### 2.2.2 Geographical Distribution

The distribution of an invasive alien plant is used as a measure to determine the impact it has on habitats (Nel *et al.* 2004:53). In the context of the Southern African Plant Invaders Atlas (SAPIA), impact is defined as the product of a species range, abundance and per capita effect (Richardson & Van Wilgen, 2004:53). *Opuntia ficas-indica* falls in the list of those plant invaders with the highest impact of occurrence as shown in Figure 2.5



**Figure 2.5** Distribution of *Opuntia ficas-indica* across South Africa (AGIS, 2007)

### 2.3 *Cereus Jamacaru*

*Cereus Jamacaru* belongs to the sub-family Cactoideae as illustrated in Figure 2.1. It is commonly known as Queen of the Night and it is native to North Eastern Brazil where it is used as cattle fodder (Do Rêgo *et al.*, 2009:34). Amongst the three subfamilies of cactaceae, Cactoidea is the most diverse and large in size (Terrazas & Arias, 2003:444).

### 2.3.1 Morphology

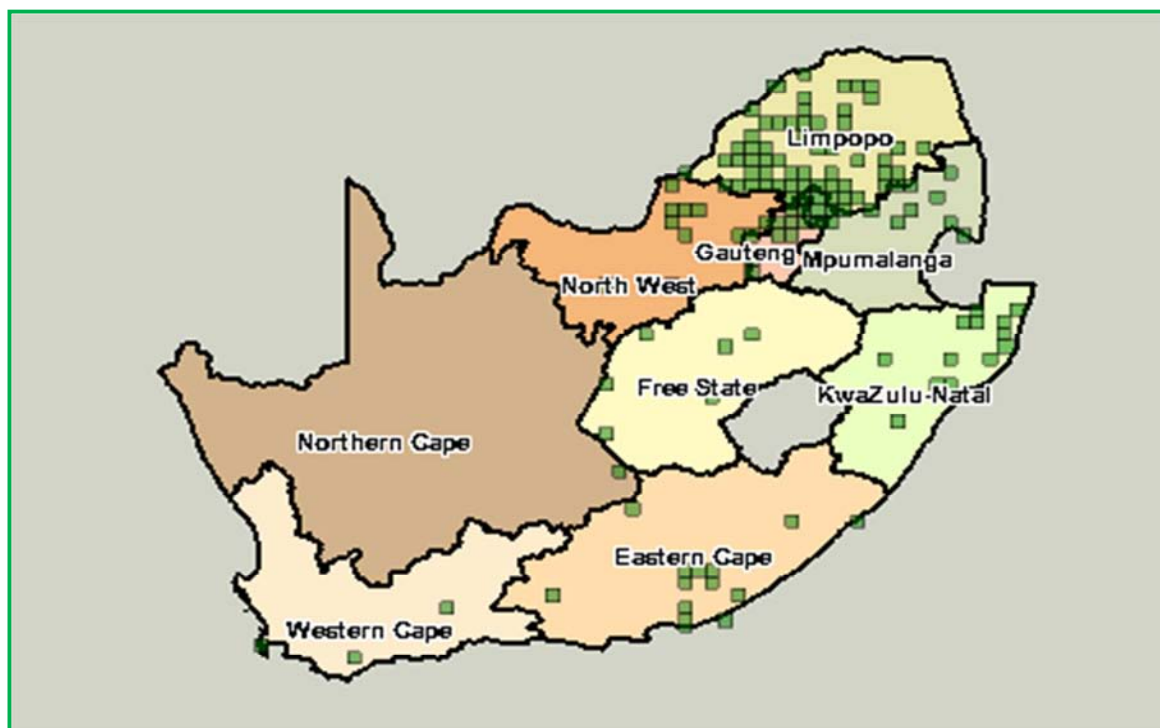
*Cereus Jamacaru* is a tree-like columnar cactus with cylindrical stems of up to 60 cm in diameter (Terrazas & Arias, 2003:445; Meiado *et al*, 2010:121). The plant grows into a dense thicket with branches growing up to 10 m (Meiado *et al*, 2010:121).The cylindrical stems are covered by spines along the 4 to 6 ribs of the plant. The plant has beautiful flowers that open up during the night, hence the name “Queen of the night”. The *Cereus Jamacaru* plant is presented in Figure 2.6 as it appears in the Vredefort Dome area.



**Figure 2.6** *Cereus Jamacaru* (Queen of the night) in the Vredefort Dome area.  
(Potchefstroom –Parys road)

### 2.3.2 Distribution of *Cereus Jamacaru*

*Cereus Jamacaru* is more sparsely distributed than *Opuntia ficas-indica* as can be seen in the distribution maps presented in Figure 2.5 and Figure 2.7, respectively.



**Figure 2.7** Distribution of *Cereus Jamacaru* across South Africa (AGIS, 2007)

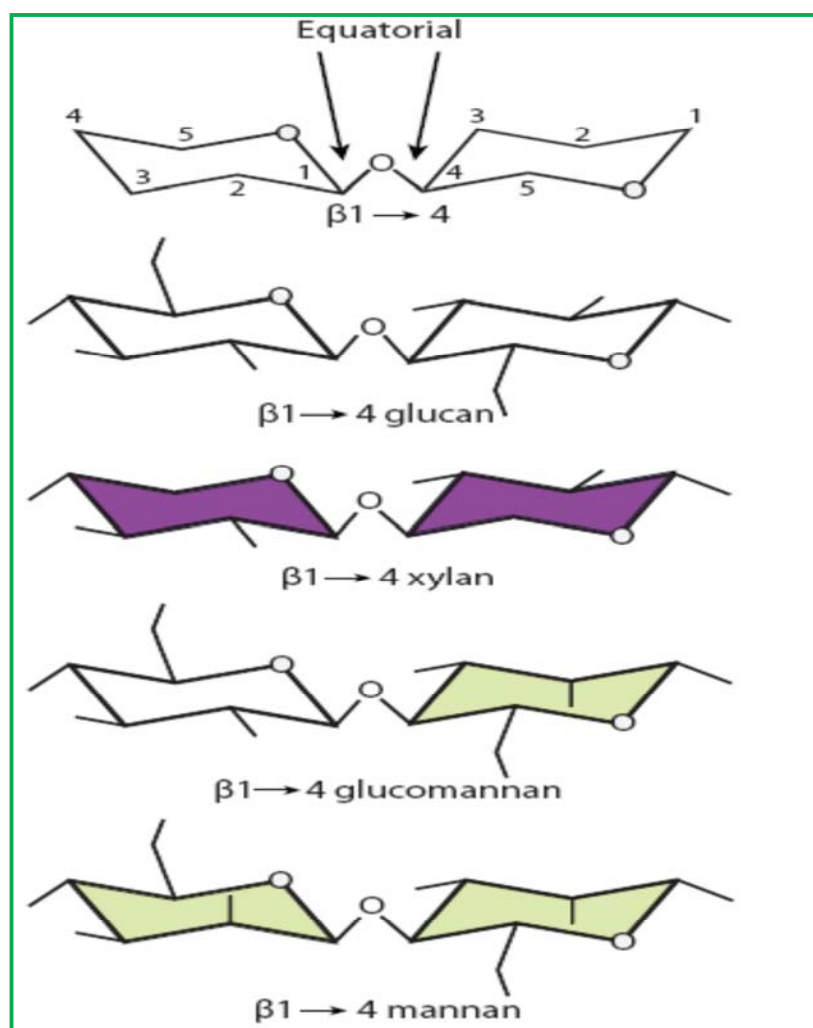
## 2.4 Polymer components of the plant cell wall

Major components of the plant cell wall relevant to this study are hemicellulose, lignin and cellulose.

### 2.4.1 Hemicellulose

Hemicellulose together with cellulose plays a major role in the strength of plant cell walls. In hard and soft wood they are interspersed with lignin (Fang *et al.*, 2000:88). Unlike cellulose, that is linear and crystalline, the structure of hemicellulose is amorphous with  $\beta$ -(1 $\rightarrow$ 4)-linked backbones and side chains that occur differ on the basis of the nature of the plant. Types of hemicellulose in plants are xyloglucans,

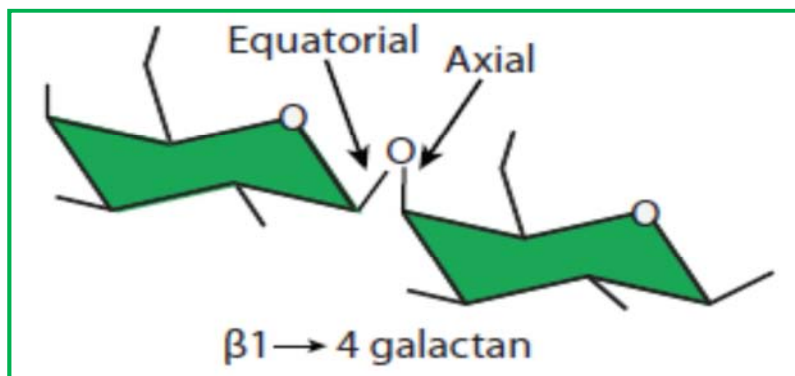
xylans, mannans, glucomannans and  $\beta$ -(1 $\rightarrow$ 3, 1 $\rightarrow$ 4)-glucans (Scheller & Ulvskov, 2010:263.) In contrast to cellulose, that has  $\beta$ -(1 $\rightarrow$ 4)-D-glucopyranosyl units only, hemicellulose is characterised by  $\beta$ -(1 $\rightarrow$ 4)-linked backbone of combination of glucose, mannose, or xylose sugars occurring equatorially at C<sub>1</sub> and C<sub>4</sub> (Scheller & Ulvskov, 2010:265) as illustrated in Figure 2.8



**Figure 2.8**  $\beta$ -(1 $\rightarrow$ 4)-linked backbone of hemicellulose showing different sugar units occurring equatorial at C<sub>1</sub> and C<sub>4</sub> (Scheller & Ulvskov, 2010: 265).

According to Scheller & Ulvskov (2010: 265) not every heteropolymer is hemicellulose. For a heteropolymer to be regarded as a hemicellulose, the sugar units must be

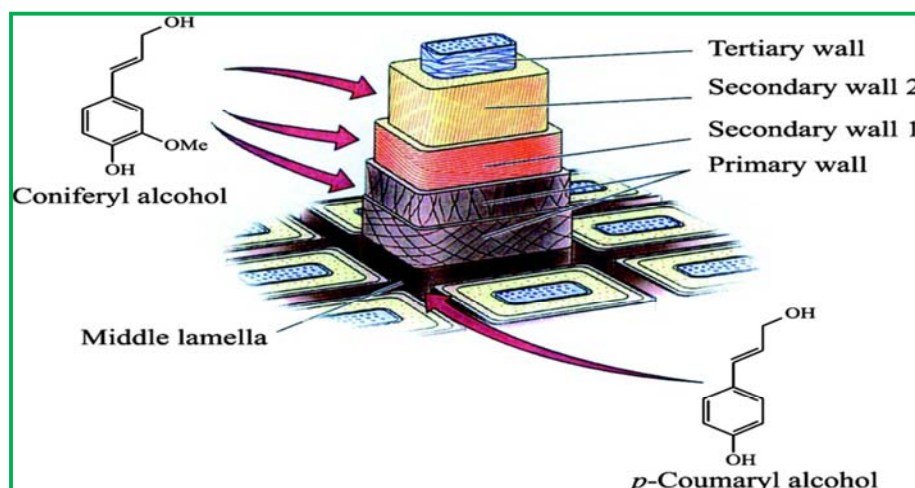
attached at the equatorial position of C<sub>4</sub> shown in Figure 2.8 and not the axial position as illustrated in Figure 2.9



**Figure 2.9**  $\beta$ -(1 $\rightarrow$ 4)-linked backbone showing axial configuration at C<sub>4</sub>  
(Scheller & Ulvskov .2010:265).

## 2.4.2 Lignin

Unpacking the structure of a substance at a molecular level is a necessary route in understanding its developmental pathway. Lignin is the second most abundant organic plant macromolecule in nature. Its molecular assembly has created much confusion among researchers for over a half a century and as a result the mechanism through which its configuration is created is still being held in two opposing views (Lewis, 1999:153).

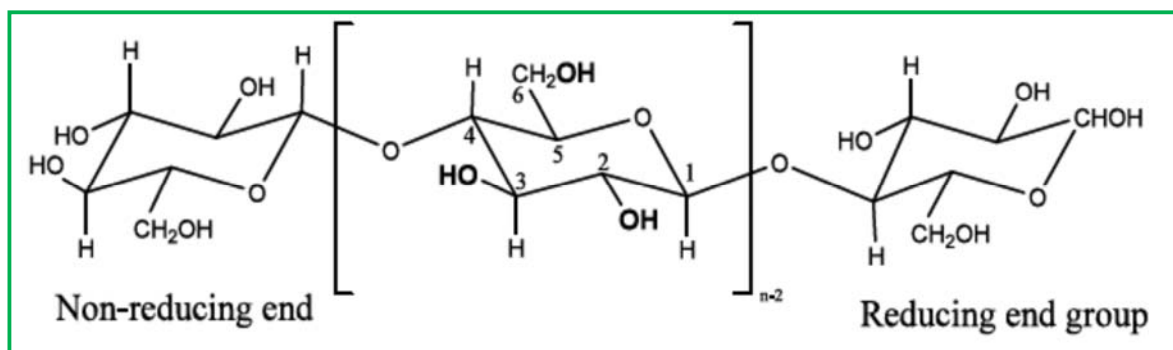


**Figure 2.10** Phenolic alcohols of lignin (Monolignols) and cell wall layers (Laurence & Lewis, 2005: 408)

Lignin is derived through free radical polymerisation (Radotić *et al.*, 1994:1763) of three phenolic alcohols (monolignols) viz. Coniferyl, *p*-coumaryl and sinapyl alcohol (Laurence & Lewis, 2005: 407; Radotić *et al.*, 1998:216) presented in Figure 2.10. Coniferyl with small amounts of *p*-coumaryl alcohols are found in woody gymnosperms and coniferyl and sinapyl alcohols with little *p*-coumaryl alcohol occur in woody angiosperms (Lewis, 1999:153).

### 2.4.3 Cellulose

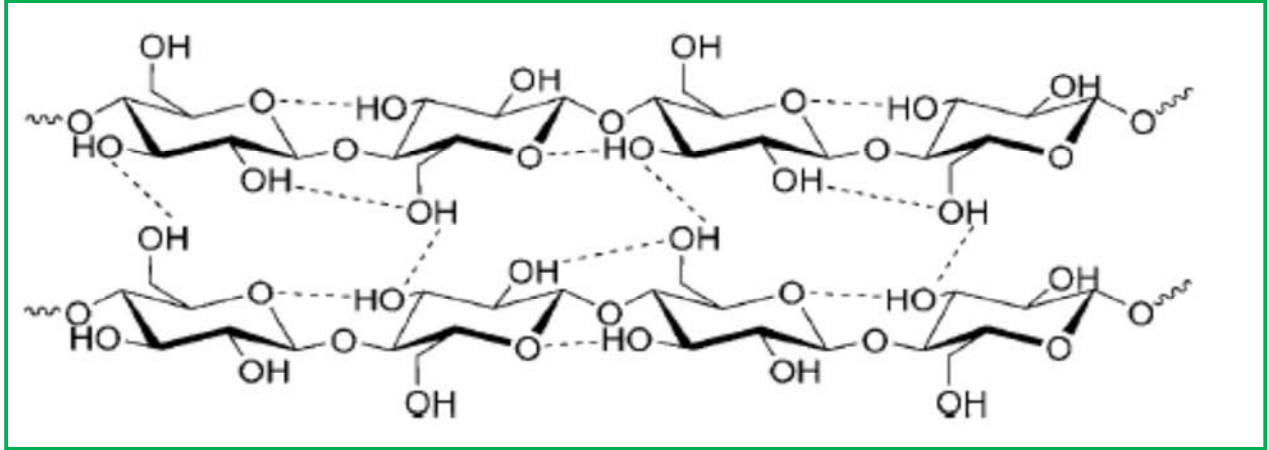
Cellulose is the main structural component of all ligno-cellulosic biomass (Pingali *et al.*, 2010: 2329). It is a linear polymer composed of  $\beta$ -D-glucopyranose (Glucose) units which are linked together by  $\beta(1\rightarrow4)$ -glycosidic bonds (Gardner *et al.*, 2008:547) between carbon C<sub>1</sub> and C<sub>4</sub> of adjacent glucose units. Each glucose unit consists of three hydroxyl groups of different reactivity attached at C<sub>2</sub>, C<sub>3</sub> and C<sub>6</sub>. Reactions on the surface of cellulose differ according to the position of the hydroxyl group. The most reactive position is C<sub>6</sub> (Primary hydroxyl) followed by C<sub>2</sub> (secondary hydroxyl) (Gardner *et al.*, 2008:546). The structure of cellulose units is illustrated in Figure 2.11.



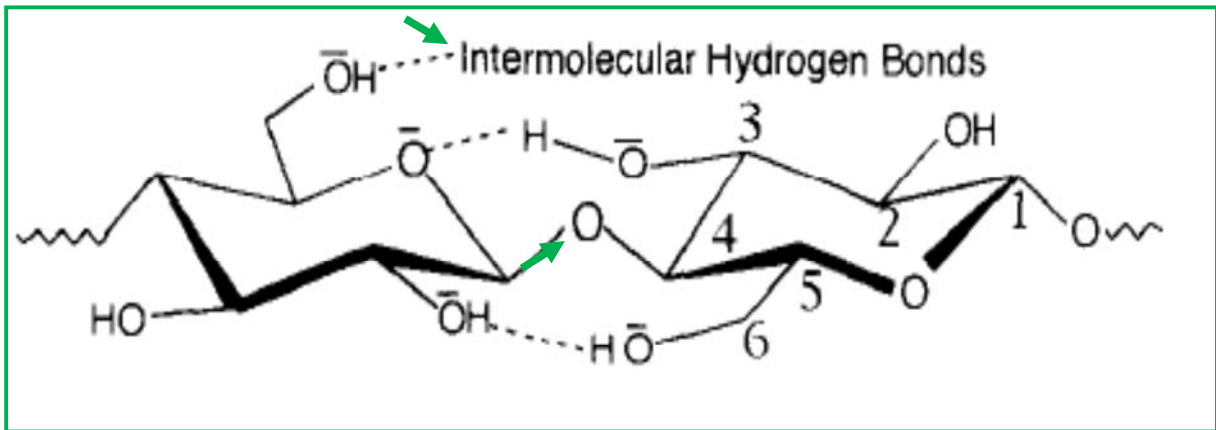
**Figure 2.11** Structure of cellulose showing glucose units linked together by (1 $\rightarrow$ 4)-glycosidic bonds (Köpcke, 2010:2).

Cellulose chains are linear and are tightly aligned parallel to each due to hydrogen bonds, forming microfibrils as illustrated in Figure 2.12 (Van de Vyver *et al.*, 2011:82; Zykwincka *et al.*, 2005:397). The hydroxyl groups on the cellulose chain form hydrogen bonds within the same chain (intramolecular) as illustrated in Figure 2.13 (Kondo &

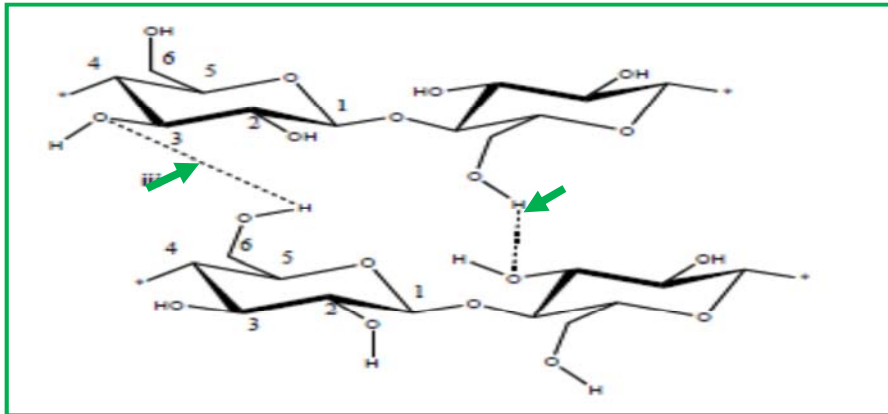
Sawatari, 1995:396) and between chains (intermolecular) illustrated in Figure 2.14 (Festucci-Buselli *et al.*, 2007:3 & Köpcke, 2010:4).



**Figure 2.12** Linear molecules of cellulose stacked together through Van der Waal's forces showing intermolecular and intramolecular hydrogen bonds (Van de Vyver *et al.*, 2011:82)

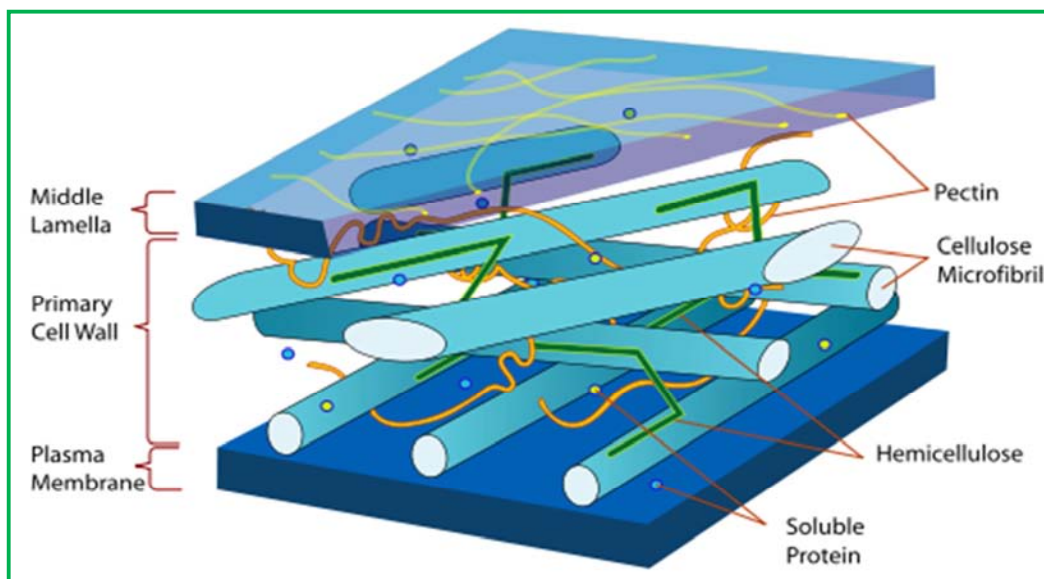


**Figure 2.13** Linear molecules of cellulose showing intermolecular hydrogen bonds (Kondo & Sawatari, 1995:396)

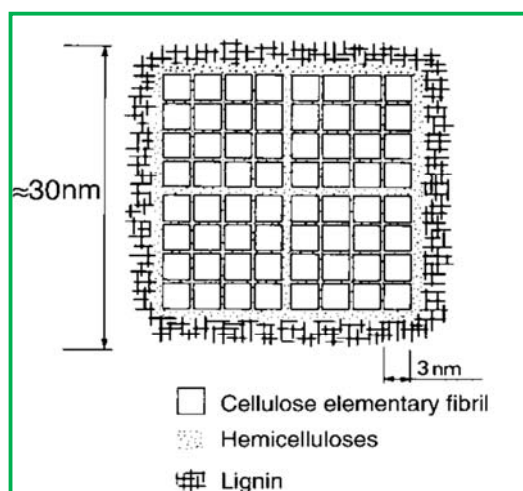


**Figure 2.14** Cellulose showing intermolecular hydrogen bonds (Köpcke, 2010:2)

Microfibrils of cellulose are interspersed within the cell wall in a network of other polymers (hemicelluloses, pectin, lignin) (Malainine *et al.*, 2005:1520) leading to the rigidity found in higher plants as illustrated in Figure 2.15 (Myllytie, 2009:7). The top view of the microfibril is presented in Figure 2.16.

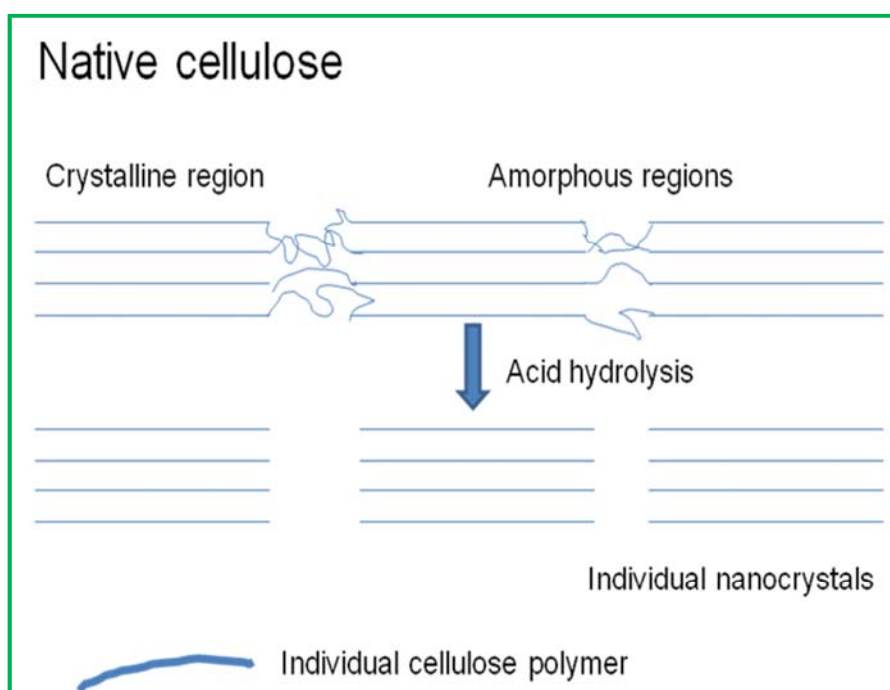


**Figure 2.15** Model of the ultra-structural organisation of the cell wall components in wood (Myllytie, 2009:7)



**Figure 2.16** Top view of cellulose microfibril with hemicelluloses and lignin (Salajková, 2009:11).

In nature cellulose occurs in two forms, *i.e.* the linear crystalline and amorphous region (Gardner *et al.*, 2008:547) as illustrated in Figure 2.17.



**Figure 2.17** Illustration of cellulose hydrolysis showing linear crystalline and amorphous region (Salajková, 2009:120).

#### 2.4.4 Isolation of cellulose

Development of materials obtained from renewable resources with the aim of producing biodegradable products has generated intensive research. The focus of research is at the abundant supply of agricultural residues (Pasquini *et al.*, 2010: 486) as well as tropical plants that include, among others, cacti (Shedbalkar *et al.*, 2010: 136). The ability of the cacti to thrive under environments understood to be stressful for most plant species distinguishes itself as a promising source of lignocellulosic material viz. cellulose, lignin, and hemicellulose (Hernández-Urbiola *et al.*, 2011:1288).

For industrial application, the components of the lignocellulosic material must be isolated and purified. Cellulose is isolated from plant material by removal of hemicellulose, lignin and other substances of the plant (Pappas *et al.*, 2002: 19; Liu *et al.*, 2006: 5742). To achieve the isolation of cellulose from within the matrix of lignin and hemicelluloses, both mechanical (Steam explosion and ultrasonic irradiation (Sun *et al.*, 2004:1712)) and chemical methods (alkaline treatment (Liu *et al.*, 2006: 5743)) are employed. In the process of the isolation of cellulose the hydrogen bonds that bind cellulose chains into crystallites and amorphous domains are disrupted and that allows easy penetration of solvent molecules into the chain molecules (Oh *et al.*, 2005: 420).

Some Industrial applications in which cellulose plays a major role (Christoffersson, 2005:11; Ververis *et al.*, 2004:246 ; Shedbalkar *et al.*, 2010:140) are:

- food ingredient for thickening, texturing and calorie reduction;
- printing paper surface coating;
- mineral processing froth-flotation depressant; and
- oil drilling and stimulation chemical.

#### 2.5 Ethanol gel

Ethanol gel is a renewable form of energy synthesised from Bio-ethanol, water and a gelling agent. It is clean, non-toxic and environmentally friendly (Darkwah *et al.*, 2008: 24). The term 'gel' was first used by Thomas Graham in 1861 (Horne, 1999:261). Since

then researchers from different fields could not come up with a common explanation as to what constitutes a 'gel'. This lack of clear description of a 'gel' (Horne, 1999:261) led Dorothy Jordan Lloyd in 1926 to state that:

*“The colloidal condition, “gel” is one which is easier to recognize than to define.”*

The indiscriminate use of the term 'gel' by scientists of different background viz. physicists, chemists, chemical engineers, biologists and medical researchers, has created ambiguity as to what a 'gel' constitutes (Almdal *et al.*, 1993:8). Their differences stem from the reference from which they define the gel. Some common reference points are rheological behaviour, structural feature, physical nature or chemical nature (Vioux *et al.*, 2010:241)

In modern terms a gel is described as a visco-elastic solid (i.e. the system which can flow like a viscous liquid on one hand and behave as an elastic solid on the other) (Horne, 1999: 261). The modern terminology incorporates the colloidal nature of a gel as advanced by D. Jordan Lloyd and the network character is described as a solvent-rich solid state made from a connected assembly of macromolecules (Almdal *et al.*, 1993:9; Dickinson & Hong, 1995: 2560). The network structures of a gel can either be covalently cross-linked in the case of chemical gels or non-covalently linked in the case of physical gels (Dickinson & Hong, 1995: 2560; Vioux *et al.*, 2010:243).

### **2.5.1 Bio-ethanol**

Bio-ethanol forms a major component of the gel synthesised in this study. The interest in bio-ethanol dates back to the 1920's but diminished in the 1960's when crude oil prices declined. The renewed interest in bio-ethanol is driven by the international pressure to go green and the impending depletion of fossil fuels (IPCC, 2007:61).

Ethanol can be produced via fermentation from any material that contains sugars. It is especially batch fermentation, *Saccharomyces cerevisiae*, that has been and is being utilised (Bahareh & Mehrdad, 2011:651). The feedstock used in the production of ethanol can be classified into three types, namely

- sugars
  - sugar cane, sugar beets, molasses, fruit
- starches
  - grains, potatoes, root crops
- cellulose materials.
  - wood, agricultural residues, waste sulfite liquor from pulp and paper mills.

Based on the feedstock, the production of ethanol can either be of first (sugars and starches) or second generation technology (lignocellulosic material) (Cardona & Sa´nchez, 2007: 2436).

### **2.5.2 Gelling agents**

The use of ethanol gel as a substitute for paraffin has gained much popularity in Southern Africa with Green Heat and Silver Sands regarded as the leading suppliers of ethanol gel in South Africa (Darkwah *et al.*, 2008: 24). Currently, gelling agents generally used in commercial gels are synthetic and many are imported ( Murdiati & Laksitoresmi, 2010:2).

## 2.6 References

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## CHAPTER 3 – EXPERIMENTAL

“One's first step in wisdom is to question everything and one's last is to come to terms with everything.” Georg Christoph Lichtenberg (1742 – 1799)

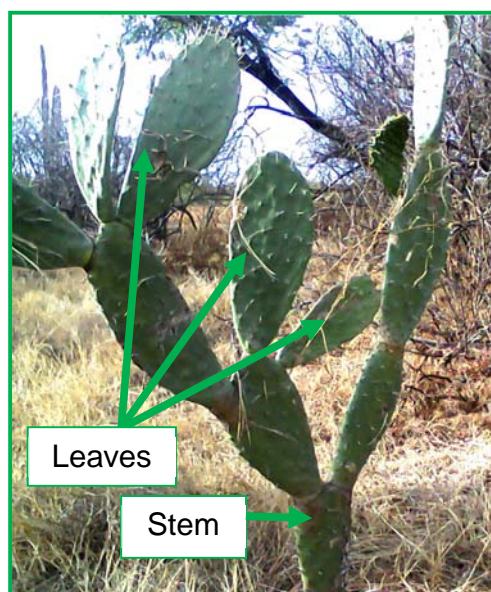
### OVERVIEW

Chapter 3 provides a detailed description of experimental work conducted. The material used in this study is presented and described in Section 3.1. In Section 3.2 the steps involved in the extraction of the gelling agents from the cacti are presented. Section 3.3 describes the synthesis of Ethanol gel. Characterisation of prepared gels is explained in section 3.4. The analytical techniques are presented in Section 3.5.

### 3.1 Materials

#### 3.1.1 Cacti species used

Two cacti species, namely *Opuntia ficas-indica* and *Cereus Jamacaru* as shown in Figure 3.1 & Figure 3.2 were used in this study. The cacti were harvested in the Vredefort Dome (Koepel) near Potchefstroom, North West Province. The stem and leaves of the *Opuntia ficas-indica* were used after their spines had been removed.



**Figure 3.1** *Opuntia ficas-indica* plant showing leaves (cladodes) and stem

The stem and leaves of *Cereus Jamacaru*, are fused, therefore the spines were removed from the fused stem.



**Figure 3.2** *Cereus Jamacaru* stems

### 3.1.2 Chemicals used

The chemicals were used as received from suppliers without any prior purification. The information about the suppliers and purity of chemicals is given in Table 3.1

**Table 3.1** Information on chemicals

Component	Purity	Supplier	Purpose
Distilled water	100.00 wt%	Oasis	Washing residue
Ethanol	99.99 wt%	Sigma-Aldrich	Synthesis of ethanol gel
Nitric Acid	98.00 N	Sigma-Aldrich	Remove lignin and oxalate crystals
Sodium Hydroxide	98.00 wt%	Fluke	Remove hemicellulose
Sodium hypochlorite	4 wt%	Ace chemicals	Remove pigments

### 3.1.3 Commercial gels

In order to determine the feasibility of the synthesised ethanol gel, it was compared with three different commercial gels (i.e. Green gel, Blue gel and Red gel). The information about suppliers is presented in Table 3.2

**Table 3.2** Commercial ethanol gel suppliers

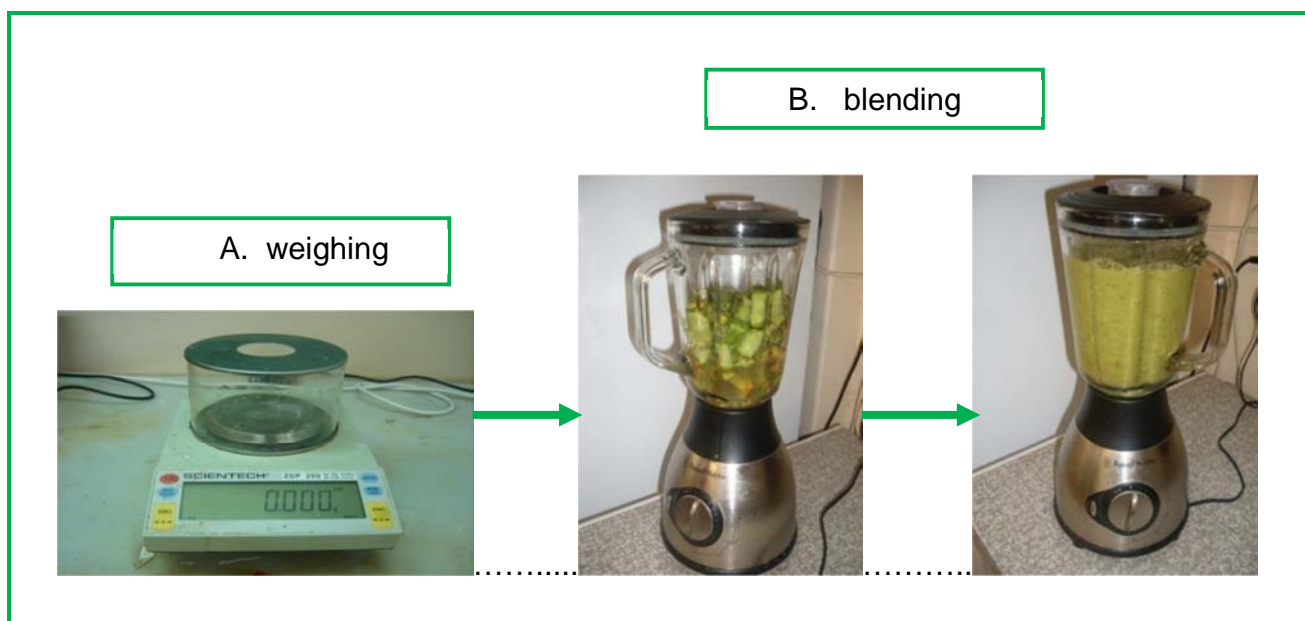
Commercial gel	Supplier
Red gel	Red Cap Gel Manufacturers
Green gel	Heat for Africa
Blue gel	Silversands Ethanol

## 3.2 Extraction of gelling agents

The aim of the investigation was to extract the cellulose from both the cacti. The leaves of *Cereus Jamacaru* are fused with the stem, so extraction was done on the stem. For *Opuntia ficas-indica*, the stem and leaves (cladodes) were used for the extraction. The extraction of cellulose was based on the method by Melainine *et al.* (2003:78).

### 3.2.1 Material and methods

Fresh cladodes of *Opuntia ficas-indica* and the fused stem of *Cereus jamacaru* were collected from the Vredefort Dome area about 10km from Potchefstroom in the North West Province of South Africa. Spines from the samples were carefully removed by a sharp and pointed knife. The cacti were washed to remove dust and dirt and then weighed on The Scientec ZSP 250 weighing balance shown in Figure 3.3. The weighed sample was cut into small pieces and added to a Russel Hobbs, 1000W Satin Blender shown in Figure 3.3. Distilled water in the ratio of 1:5 was added and the cacti blended to a slurry.

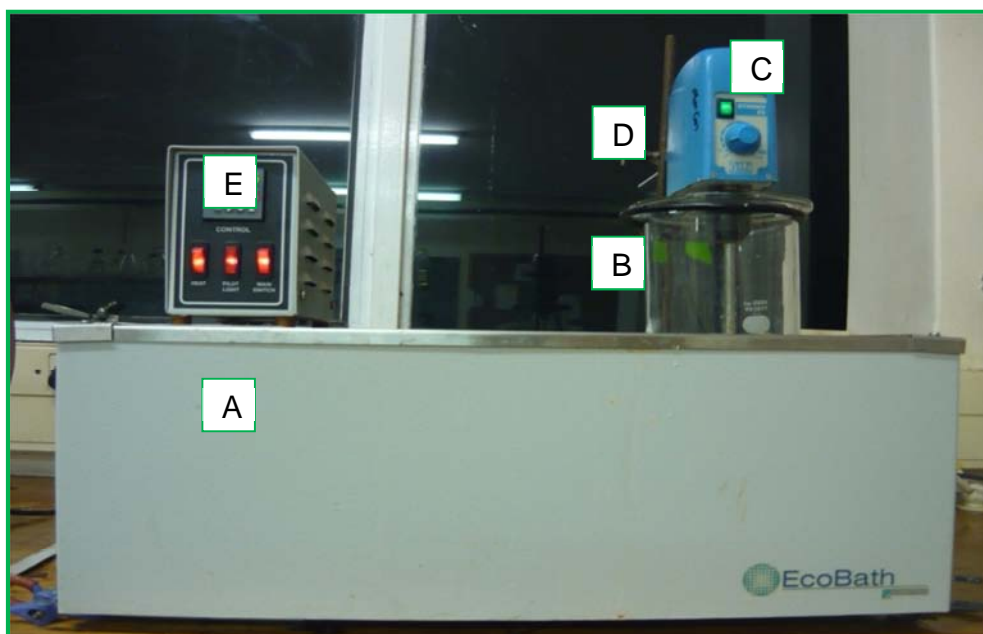


**Figure 3.3** Weighing and blending of diced cacti

[A. Scintec ZSP 250 weighing balance B. Russel Hobbs, 1000W Sation Blender]

### 3.2.2 Extraction process

The method of Melainine *et al.* (2003:78) was followed with slight modification. The isolation of cellulose was done on samples of *Opuntia ficas-indica* stem and leaves as well as the sample of fused *Cereus Jamacaru* stem. The extraction process was done at set temperatures using the water bath system shown in Figure 3.4



**Figure 3.4** Water baths showing overhead stirrer and digesting vessel  
[A. Water bath B. 4L Pyrex glass vessel C. Overhead stirrer D. Retort stand  
E. Heating unit]

### 3.2.2.1 Water digestion

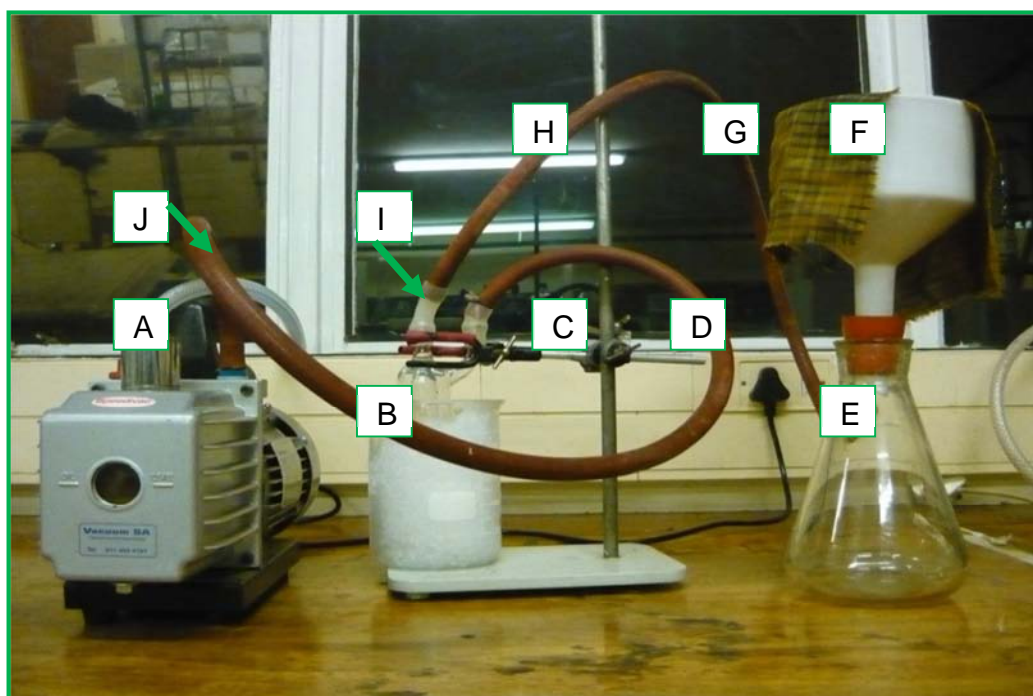
Samples (600g) were blended into a slurry, deposited into a 4 L Pyrex glass vessel (B) and then suspended in distilled water (3000 ml) for 2hrs at 50 °C with constant stirring using an overhead stirrer (C). Every extraction step was followed by vacuum filtration as shown in Figure 3.5

### 3.2.2.2 Alkali extraction

After 2 hrs of water extraction, the water insoluble residue was filtered with a cotton cloth (G) and dispersed in a 2% NaOH solution. The alkali suspension was digested for 2 hrs at 50 °C, filtered and extensively washed with distilled water. Washing the alkali-insoluble product with water remove soluble polysaccharides and calcium oxalate crystals (Melainine *et al.*, 2003:78).

### 3.2.2.3 Bleaching

The alkali-insoluble residue was bleached with 2.5% Sodium Hypochlorite. Bleaching was carried out at 60 °C for 2 hrs. The bleaching process remove most of the residual lignin and proteins (Melainine *et al.*, 2005:1521).



**Figure 3.5** Filtering system connected to a vacuum pump

- [A. Vacuum pump B. Cooling jacket C. Retort stand D. Hose to vacuum flask  
E. Buchner flask F. Buchner funnel G. Filtering cotton cloth H. Hose from  
Buchner flask I. Dropping flask J. Hose from vacuum pump]

### 3.2.2.4 Nitric acid digestion

The bleached residue was subsequently treated with 0.05N Nitric acid solution treatment at 60 °C for 1 hr. The residue obtained from Nitric the Acid treatment was washed with distilled water to remove residual oxalate crystals. The Nitric Acid treated residue was dried to a semi-dry pulp in a vacuum oven shown in Figure 3.6 which was set at 80 °C for 40 min. The semi-dry pulp was analysed for moisture using a HR83 Halogen Moisture Analyser shown in Figure 3.7 in order to calculate the yield of cellulose on dry basis.



**Figure 3.6** Vacuum oven for drying samples

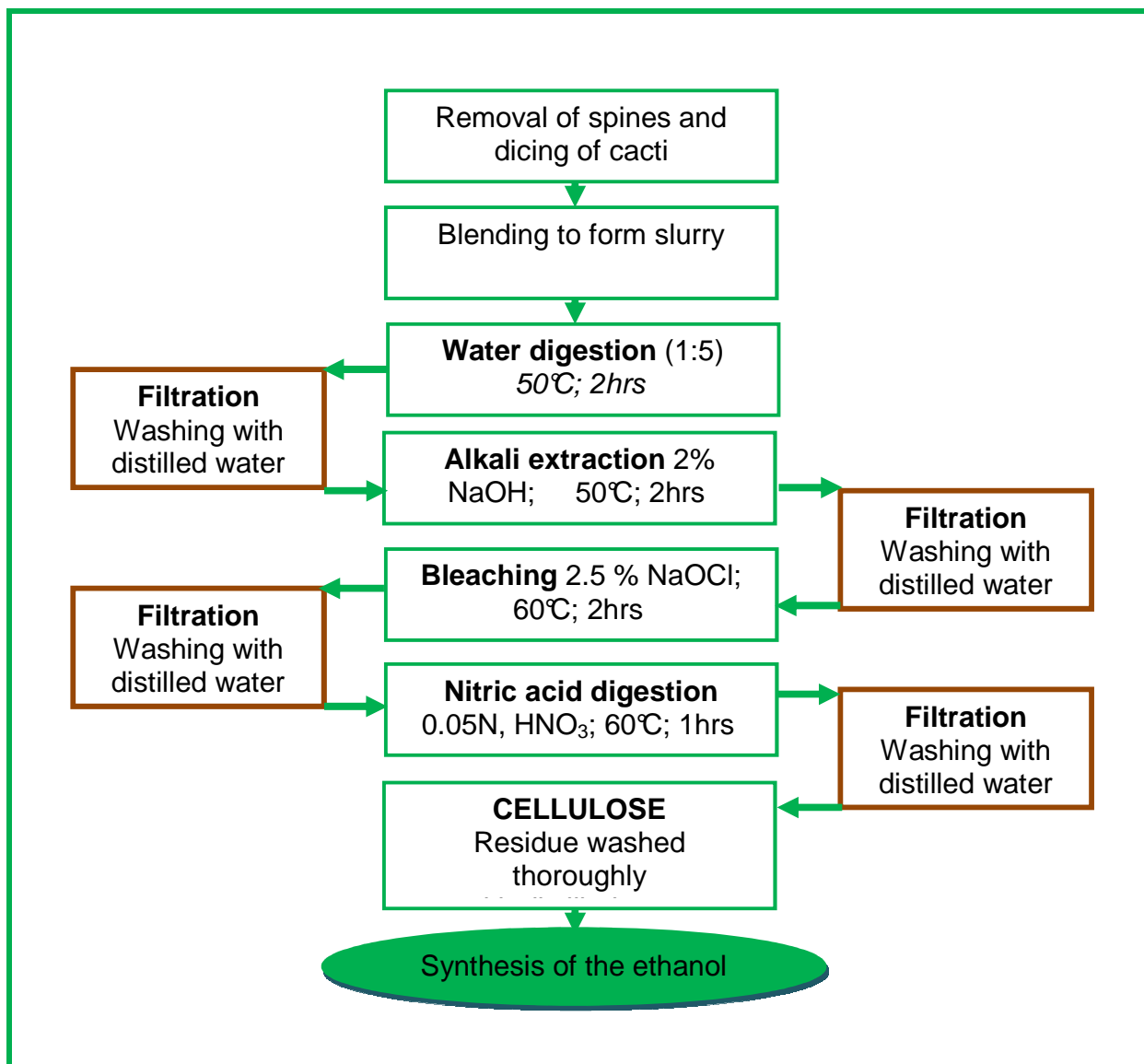


**Figure 3.7** HR83 Halogen Moisture Analyzer

The pulp obtained after vacuum drying was semi-dry and it was used for the synthesis of ethanol gel.

### 3.2.3 The Extraction Process Flow Diagram

The extraction process is presented in a flow diagram illustrated in Figure 3.8 showing conditions for each step.



**Figure 3.8** Flow diagram of Extraction process of cellulose

### 3.3 Synthesis of Ethanol Gel

Ten ethanol gels of different compositions were prepared from both *Opuntia ficas-indica* stem and *Cereus Jamacaru stem*. The gels were synthesised from Bio-ethanol, water and a gelling agent. The compositions were such that ethanol should not be less than

80% by weight. The other 20% consisted of water and cellulose (SABS, SANS 448:2010). Five different compositions for each cactus were prepared from cellulose obtained from both *Opuntia ficas-indica* and *Cereus Jamacaru* stems. Gels of *Opuntia ficas-indica* were denoted, *Opuntia 6, 9, 12, 15, 18* and the gels of *Cereus Jamacaru*, *Cereus 6, 9, 12, 15, 18*.

The semi dry cellulose described in Section 3.2.2 with a moisture content of 65% was used in the synthesis of the ethanol gels. The components of each gel viz. water, cellulose and ethanol were mixed to a total of 120g by weight as illustrated in Table 3.3. In producing a gel, water, cellulose and ethanol were blended in a Russell Hobbs, 1000W Satin Blender. Homogenisation of cellulose in the presence of ethanol and water led to the disruption of hydrogen bonds that bind the cellulose micro fibrils allowing access of the solvents and thus initiating gelling (Kim *et al.*, 2002:193). As the suspension was blended the viscosity increased. Blending was carried out until there was no further increase in viscosity of the suspension. The prepared gels with their respective compositions are presented in Table 3.3 and Table 3.4

**Table 3.3** Different gels of *Opuntia ficas-indica* prepared with varying proportions of Cellulose, Ethanol and Water. (SANS 448.2010:4)

<b>Gel type</b>	<b>% Cellulose</b>	<b>% Water</b>	<b>% Ethanol</b>
Opuntia 6	1.750	3.250	95.0
Opuntia 9	2.625	4.875	92.5
Opuntia 12	3.500	6.500	90.0
Opuntia 15	4.375	8.125	87.5
Opuntia 18	5.250	9.750	85.0

**Table 3.4** Different gels of *Cereus Jamacaru* prepared with varying proportions of Cellulose, Ethanol and Water (SANS 448.2010:4).

<b>Gel type</b>	<b>% Cellulose</b>	<b>% Water</b>	<b>% Ethanol</b>
Cereus 6	1.750	3.250	95.0
Cereus 9	2.625	4.875	92.5
Cereus 12	3.500	6.500	90.0
Cereus 15	4.375	8.125	87.5
Cereus 18	5.250	9.750	85.0

### 3.4 Characterisation of Ethanol Gels

In order to characterise the prepared ethanol gels, their burn time, waste (residue), viscosity and caloric values were determined.

#### 3.4.1 Burn time

Burn time is the time it takes for the gel to burn to completion without any external intervention. Of each of the gels 5.00g was placed in a heat resistant beaker and set on fire. A stop watch was used for taking the time for the complete combustion of the gel

#### 3.4.2 Waste or residue

The residue or waste that remains after complete combustion of the gel.

#### 3.4.3 Viscosity

Viscosity of a gel is a property of offering resistance to the non-accelerated displacement of two adjacent layers. In order to measure the viscosity, the samples were blended at different times and their viscosity measured by using a Brookfield viscometer shown in Figure 3.9. For all the samples the spindle size R3 was used and the rotation maintained at 100 Rpm.

### 3.4.4 Calorific value

The calorific value of the prepared gel was analysed by the Agricultural Research Council's (ARC).

## 3.5 Equipment for Analyses and Methodology.

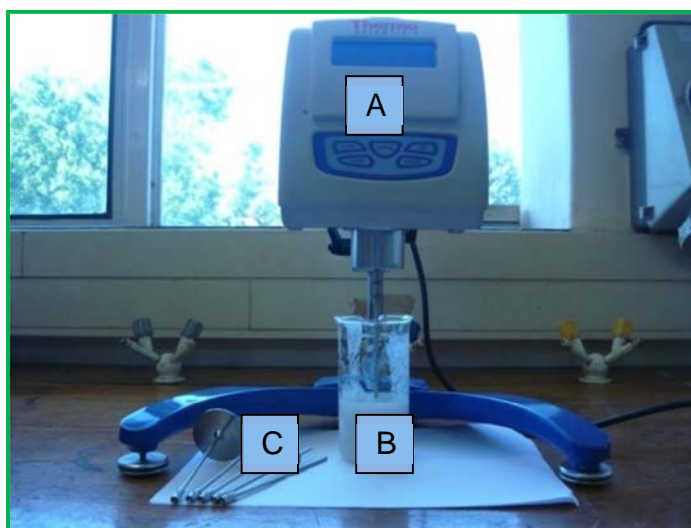
In this section a description of the equipment used in the study is presented. A discussion about FTIR is given in Section 3.5.1 and Section 3.5.2 describes the Brookfield viscometer.

### 3.5.1 Fourier transform infrared (FT-IR)

Infrared spectroscopy (FT-IR) was used to characterise the chemical modifications of the samples after each treatment during the extraction process as explained in Section 3.2.2. The spectra are expressed as percentage transmittance versus wave number ( $\text{cm}^{-1}$ ). The principle of FT-IR is discussed in Appendix C.

### 3.5.2 Brookfield viscometer

The viscosities of the gels were measured by a Brookfield viscometer using spindle R3 at room temperature. The Viscometer was set at 100 Rotations per minute (Rpm)



**Figure 3.9** Brookfield DV-II viscometer with set of spindles  
[A. Viscometer B. Ethanol gel sample C. Set of spindles]

### 3.6 References

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## CHAPTER 4 - RESULTS AND DISCUSSION

*“I count him braver who conquers his desires than him who conquers his enemies, for the hardest victory is the victory over self.” Aristotle.*

### OVERVIEW

In this chapter all the results obtained in this study according to the experimental methods described in Chapter 3 are presented. The experimental errors are presented in Section 4.1. Constituents and chemical composition is presented in Section 4.2. The Characterisation of Extracted Cellulose is discussed in Section 4.3 and Characterisation of the Synthesised Ethanol Gel is presented in Section 4.4.

#### 4.1 Experimental Error and Repeatability

The experimental error was determined for a 95% confidence level (assuming a normal distribution of yield values). Appendix A.2 provides a detailed description of the calculations of the experimental error. In order to determine the experimental error for the extraction of cellulose an experiment was repeated three times under the same operating conditions. Operating conditions under which the experiments were conducted are presented in Table 4.1.

**Table 4.1** Conditions for the determination of the experimental error

	<b>Water digestion (Water)</b>	<b>Alkaline extraction (NaOH)</b>	<b>Bleach (Sodium hypochlorite)</b>	<b>Nitric digestion (Nitric acid)</b>
Time period for the extraction(hr)	2	2	2	1
Reaction temperature(°C)	50	50	60	60
Reagent strength	distilled	2 wt%	2.5 wt%	0.05N
Reagent volume(ml/600g fibre)	3000	2500	2000	2000

The experimental errors as calculated for the yields are 6.7% for *Opuntia ficas-indica* stem, 6.8% for *Opuntia ficas-indica* leaves and 7.8% for *Cereus Jamacaru* stem. The experimental errors obtained for the extraction of cellulose from the *Opuntia ficas-indica* stem, and the *Opuntia ficas-indica* leaves suggest that the process produces consistent yields of cellulose. The difference in the experimental error obtained for the extraction of cellulose from *Cereus Jamacaru* stem and *Opuntia ficas-indica* stem, may be attributed to the presence of a greater amount of mucilage in the stem of *Cereus Jamacaru*. Table 4.2 presents the experimental errors and the yields of cellulose.

**Table 4.2** Calculated experimental errors for the extraction of cellulose

	Average yield (%)	Standard deviation	Error at 95% Confidence level
<i>Opuntia ficas-indica</i> stem	15.0	0.9	6.7
<i>Opuntia ficas-indica</i> leaves	12.7	0.8	6.8
<i>Cereus Jamacaru</i> stem	11.5	0.6	7.8

#### 4.2 Constituents and chemical composition

Plant material consists of solubles that include starches, proteins, sugars and indigestible fibre commonly known as detergent fibre that provides structural support to the plant. In determining the composition of plant fibre, three different detergent fibres were measured viz. Neutral detergent fibre (NDF), Acid detergent fibre (ADF) and Acid detergent lignin (ADL) (Van Soest *et al.*, 1991: 3584). ADF (principally lignin and cellulose) decreases as the cladodes mature (Rodriquez-Felix & Cantwell, 1988: 88). The decrease of ADF occurs as a result of the proportional increase of water storing parenchyma in relation to the outer photosynthetic parenchyma tissue (Rodriquez-Felix & Cantwell, 1988: 88). The percentage Cellulose, Hemicellulose and Lignin of *Cereus Jamacaru* and *Opuntia ficas-indica* were determined using Equations 4.1 to 4.3.

$$\text{Percentage (\% cellulose)} = ADF - ADL \quad (4.1)$$

$$\text{Percentage (\% hemicellulose)} = NDF - ADF \quad (4.2)$$

$$\text{Percentage (\% lignin)} = ADL \quad (4.3)$$

The results of *Cereus Jamacaru* and *Opuntia ficas-indica* compositions analysed by the Agricultural Research Council (ARC) are presented in Table 4.3.

**Table 4.3** The constituents and chemical compositions of *Cereus Jamacaru* and *Opuntia ficas-indica* before and after purification.

Sample	Cellulose ADF-ADL (wt%)	Hemicellulose NDF-ADF (wt%)	Lignin ADL (wt%)
<b><i>Cereus Jamacaru</i> stem</b>			
Untreated fibre	21.85	20.62	3.69
Water extraction	25.96	21.16	3.47
Bleach	26.20	14.20	3.92
Alkaline extraction	26.94	11.24	4.15
Nitric extraction	39.78	7.14	3.38
<b><i>Opuntia ficas-indica</i> leaves</b>			
Bleach	38.01	2.39	4.22
Nitric extraction	45.90	1.50	6.53
<b><i>Opuntia ficas-indica</i> stem</b>			
Untreated fibre	26.23	20.05	2.87
Water extraction	21.93	26.73	2.41
Bleach	26.37	5.11	5.06
Alkaline extraction	30.84	1.64	4.48
Nitric extraction	56.70	0.75	2.57

High cellulose content of untreated *Opuntia ficas-indica* stem (26.23 wt%) compared to cellulose content of untreated *Cereus Jamacaru* fibre (21.85 wt%) is observed. Melainine *et al.* (2003:79) reported a cellulose content of 21.6 wt% for untreated fibre of *Opuntia ficas-indica* which is lower than that obtained in this study. The lignin content of *Cereus Jamacaru* (3.69 wt %) is higher than the lignin content of *Opuntia ficas-indica* stem (2.87 wt%). Both *Cereus Jamacaru* and *Opuntia ficas-indica* show comparable hemicellulose content, viz. 20.62 wt% and 20.05 wt% respectively.

Cellulose and hemicellulose are characterised by strong interactions developed during the assembly of the cell wall (Habibi *et al.*, 2008:102). As the hemicellulose is solubilised, during the purification process, the increase in cellulose content is observed. Both *Cereus Jamacaru* and *Opuntia ficas-indica* show an increasing trend as illustrated in Table 4.3. The same trend is reported by Melainine *et al.* (2003:79) as shown in Table 4.4

**Table 4.4** The composition of cladodes of *Opuntia ficas-indica* as reported by Melainine *et al.* (2003:79)

<i>Opuntia ficas-indica</i>	Cellulose	Other polysaccharides	Lignin
Raw fibre	26.23	48.0	2.57
Alkaline extraction	30.84	10.05	X
Nitric extraction	56.70	15.1	X

[X - value not provided]

Elimination of hemicellulose from the cacti cell wall is limited by a lignin network interspersed within the cell wall fibres. Hemicellulose and lignin are associated within the cell wall matrix by ester and ether linkages (Sun *et al.*, 2004:379). After bleaching and alkali extraction there is a considerable reduction of hemicellulose from *Opuntia ficas-indica* stem (20.05 wt% to 1.64 wt %) compared to the reduction of hemicellulose from *Cereus Jamacaru* (20.62 wt% to 11.24 wt %). The reduction of hemicellulose from *Opuntia ficas-indica* stem and *Cereus Jamacaru* indicates the strength of the lignin-hemicellulose association in both cacti. Less hemicellulose is liberated from the *Cereus Jamacaru* cell wall matrix when compared to *Opuntia ficas-indica*. The smaller amount of hemicellulose liberated from the *Cereus Jamacaru* cell wall matrix can be attributed to the strength of hemicellulose and lignin linkages. The yield of *Opuntia ficas-indica* stem ( $15 \pm 6$  wt%) is higher than the yield of *Opuntia ficas-indica* leaves ( $12.7 \pm 6$  wt %) and *Cereus Jamacaru* stem ( $11.5 \pm 7$  wt %). The difference in the yield between *Opuntia ficas-indica* and *Cereus Jamacaru* compliments the amount of hemicelluloses removed from both cacti.

### 4.3 Characterisation of cellulose

Fourier Transform Infrared Spectroscopy (FT-IR) was used to examine structural changes in the cellulose fibres. FT-IR is a non-destructive method used to confirm the changes in the composition of the extracted cellulose (Oh *et al.*, 2005:424). The FT-IR spectra of each sample were recorded on a Bruker, Tensor 27 spectrometer equipped with the diamond ATR software (Opus version 6.5) data analysis and the outputs were collected in the range of 4000-400  $\text{cm}^{-1}$  and recorded in transmittance mode as a function of wave number ( $\text{cm}^{-1}$ ). The characteristic absorption bands of functional groups are presented in Table 4.5. Correlation of absorption frequencies in Table 4.5 to frequencies of extracted fibre assist in identifying the removal or retention of fibre components (i.e. Hemicellulose and Lignin). Representative absorption bands may differ in terms of intensity, shape and position ( $\text{cm}^{-1}$ ). The intensity of the absorption band may be weak, medium or strong and the shape either broad or sharp.

**Table 4.5** The characteristic absorptions and their possible assignments

	Assignment	Absorptions ( $\text{cm}^{-1}$ )	References
1	O–H stretching vibration	3200 - 3500	Yan Yan <i>et al.</i> , 2009:61
2	C– H stretching vibration	2,800 - 2,900	Yan Yan <i>et al.</i> , 2009:61
3	-CH <sub>2</sub> bending bond due to hydrogen bonds in cellulose.	1431	Sun <i>et al.</i> , 2007:2388
4	C–O stretching vibration of cellulose	1014	Sun <i>et al.</i> , 2004:1717; Sun <i>et al.</i> , 2007:2388.
5	C–H rock vibrations of cellulose	879	Sun <i>et al.</i> , 2007:2388; Sun <i>et al.</i> , 2004:1717.
6	ester carbonyl (C=O) group (Hemicellulose)	1735–1750	Sun <i>et al.</i> , 2004:1717.
7	C–H deformation vibration of lignin,	1507	Sun, <i>et al.</i> , 2004:1717.

The FT-IR spectra of the fibre extracted by alkaline and nitric solutions are illustrated in Figure 4.1 to Figure 4.4.

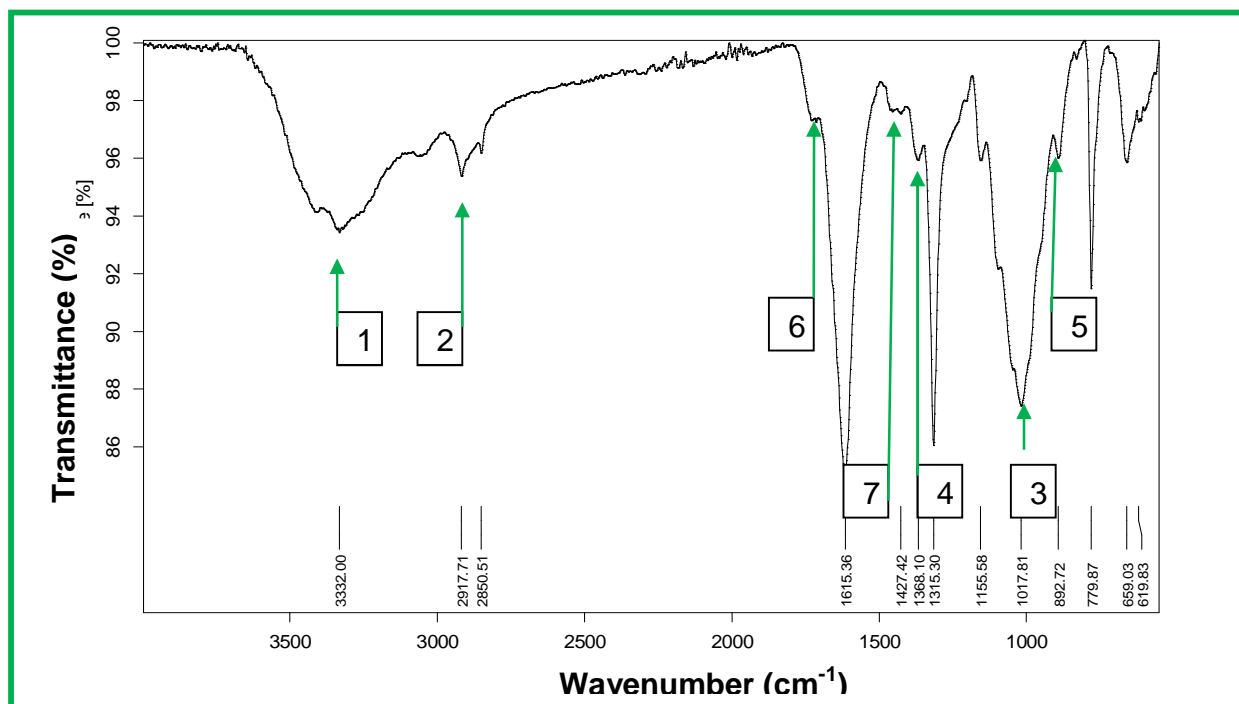


Figure 4.1 *Opuntia ficus-indica* – Alkaline treated

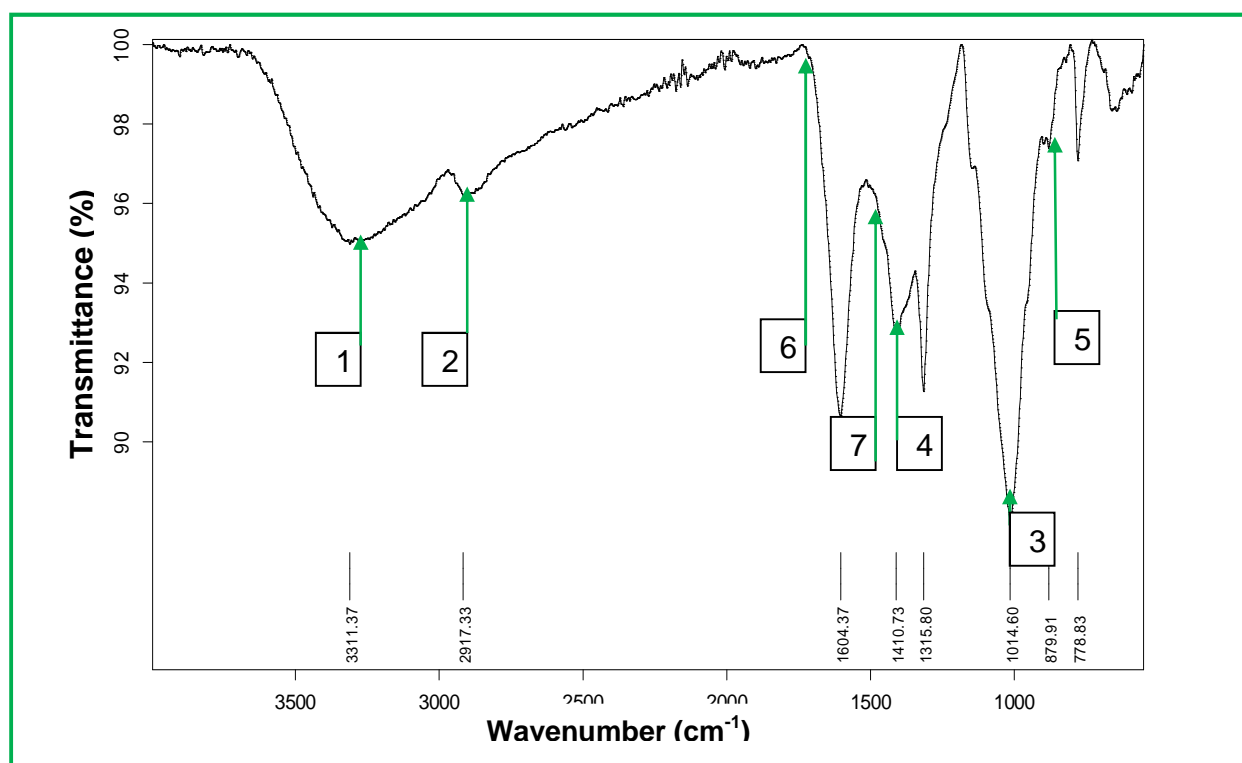
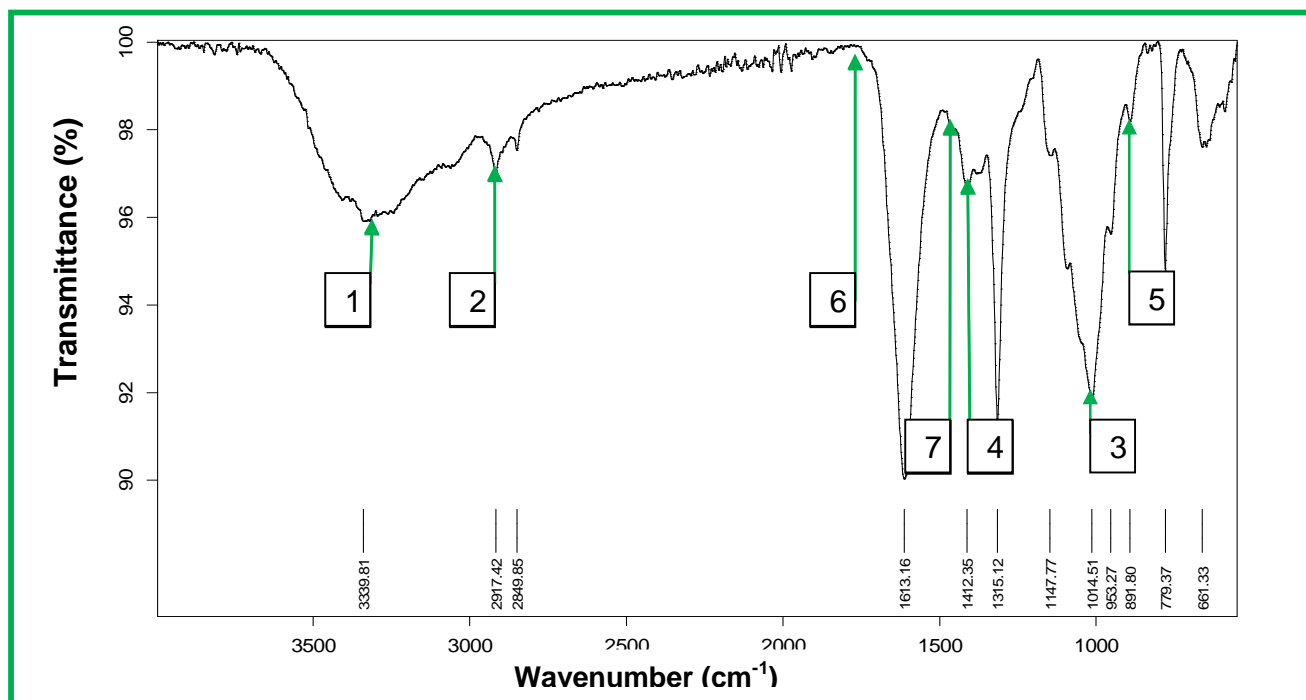


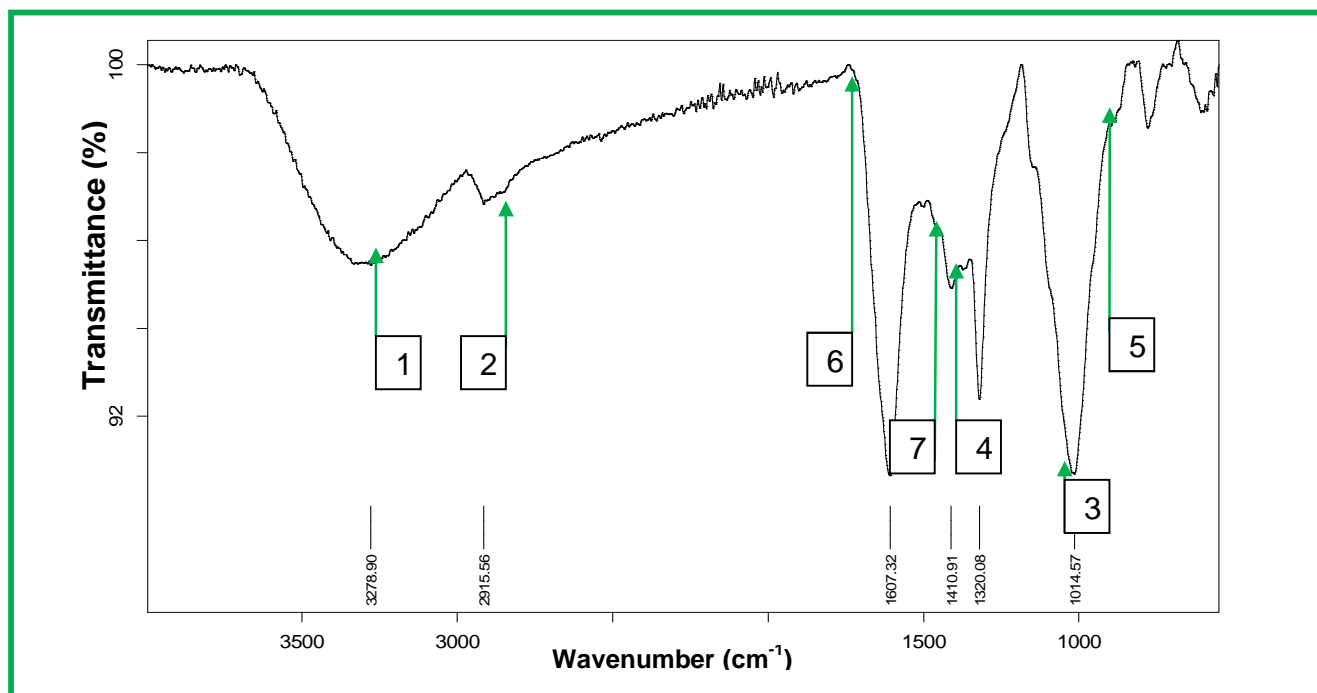
Figure 4.2 *Opuntia ficus-indica* – Nitric acid treated

The band at 3200-3500  $\text{cm}^{-1}$  (1) is attributed to the hydrogen bonded O-H stretching vibration (Adel *et al.*, 2010: 4449). The increase in the intensity of O-H vibration band (1) in Figure 4.2 indicates liberation of hydroxyl groups on the cellulose backbone as hemicellulose and lignin are removed by nitric acid digestion (Freire *et al.*, 2005:454; Adele *et al.*, 2010:4449). The peak at 1507  $\text{cm}^{-1}$  (7) represents C-H deformation vibration of lignin (Sun *et al.*, 2004:1717). The intensity of the peak at 1507  $\text{cm}^{-1}$  (7) is very weak at the alkaline treated fibre and totally absent after nitric acid treatment, showing a gradual to total removal of lignin (Alemdar & Sain, 2008: 1668). A small band at 1718  $\text{cm}^{-1}$  (6) is associated with the carbonyl (C=O) stretching unconjugated ketones (Sun *et al.*, 2004: 1717). Carbonyls occur in lignin's structural units, as explained in Section 2.4.2 as part of side chains. The absence of C=O band (6) in Figure 4.2 shows that lignin was eventually removed from *Opuntia ficas-indica* fibre by nitric acid treatment (Yan Yan *et al.*, 2009:454). Hydrogen bonds of cellulose have influence on the bending of  $-\text{CH}_2$  bond (Kondo & Sawatari, 1995:396). This explains the shift in position of the band at 1427  $\text{cm}^{-1}$  (4) in Figure 4.1 to 1410  $\text{cm}^{-1}$  (4) in Figure 4.2 and 1412  $\text{cm}^{-1}$ (4) in Figure 4.3 to 1410  $\text{cm}^{-1}$  (4) in Figure 4.4. The shift of C-H stretching vibration frequency band from high to low positions occurs as the result of the weakening of the Hydrogen interactions. (Zhang *et al.*, 2010: 955). The weakening of hydrogen interactions can be attributed to the removal of hemicelluloses and the disruption of the crystallinity of cellulose bundles (microfibrils) (Yang & Wyman, 2004:93; Taherzadeh & Karimi, 2008:1639).



**Figure 4.3** *Cereus Jamacaru* - Alkaline treated

The prominent band at  $1017\text{ cm}^{-1}$  (3) in Figure 4.1 appears in all spectra at slightly different position (i.e.  $1014\text{ cm}^{-1}$  (3) in Figure 4.2 to Figure 4.4). It is attributed to C-O-C pyranose ring skeletal vibration but has been reported at higher wavenumber than observed in this study by Sun and co-workers (2004:387). The difference stem from the fact that different plant species are used. The intensity of this peak increases from the alkaline treated fibre to nitric acid treated fibre, showing that cellulose fibres are better exposed after both lignin and hemicelluloses have been removed.



**Figure 4.4** *Cereus Jamacaru* - Nitric acid treated

The peaks at  $1718\text{ cm}^{-1}$  (6) and  $1507\text{ cm}^{-1}$  (7) indicate that the presence of hemicellulose and lignin differ in their intensity for both cacti. After nitric acid treatment the peaks occur as small shoulders for *Cereus Jamacaru* shown in Figure 4.4, whereas in *Opuntia ficas-indica* they disappear completely as illustrated in Figure 4.2. This indicates that most of the hemicelluloses and lignin were removed from *Opuntia ficas-indica* than from *Cereus Jamacaru* during the extraction process.

#### 4.4 Characterisation of ethanol gels

In order to characterise the prepared ethanol gels, the waste residue (ash content), burn time, viscosity and caloric values were determined and compared with those of commercial gels as presented in Table 4.6. Figure 4.5 illustrates the effect of cellulose content on the residue of the gels. The rate of burning and the viscosity are presented in Figure 4.6 and Figure 4.7, respectively.

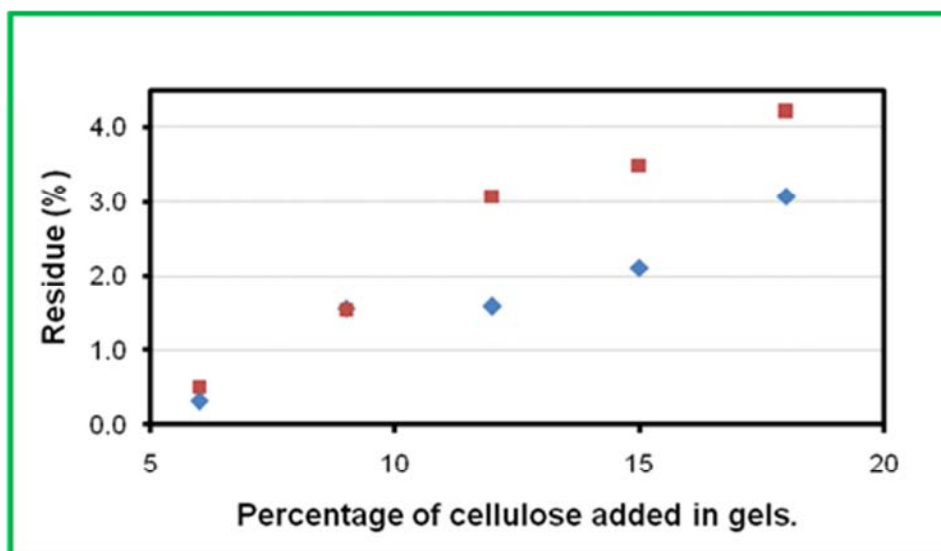
**Table 4.6** Properties of commercial gels and Paraffin

	Green gel	Blue gel	Red gel	Paraffin
Burn time (s)	108.667	113.00	92.67	86.67
Gel time (g/min)	2.62	2.41	3.28	5.73
Viscosity (100 Rpm, R3) cp	10670	19020	15370	-
Ash content (Residue (%))	6.09	9.49	0.21	1.65
Calorific values (KJ/100g)	14.88	24.07	10.81	44.7 <sup>a</sup>

[a- Lloyd & Truran, 2008:3]

#### 4.4.1 Residue of the gels

As the amount of extracted cellulose added as gelling agent increases, the ash content (Residue) left after burning the gels increased. *Opuntia* gel at 18% cellulose, has a residue of 3.07 % residue and *Cereus* gel at the same cellulose content has a residue of 4.2 %. The highest residues of the prepared gels shown in Figure 4.5 are still lower than those of commercial gels i.e. Green gel (6.09 %) and Blue gel (9.49%) presented in Table 4.6.



**Figure 4.5** Effect of cellulose content on the ash content of the gels

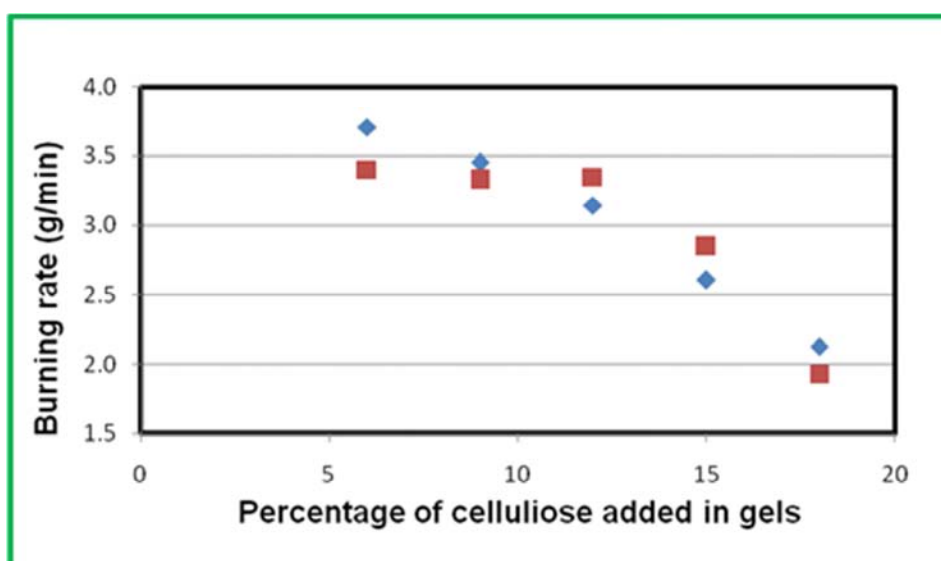
[ ■ - *Cereus Jamacaru* gel, ◆ - *Opuntia ficas-indica* ]

The *Cereus* gel residue of 4.2% is the highest of the prepared gels at less than 5% as prescribed by the South African Bureau of Standards (SANS 448. 2010:5).

#### 4.4.2 Burning rate

The Burning rate can be described in terms of heat release rate, mass loss rate or generally as the rate at which a given material is consumed by fire (Tran & White. 1992: 197). It can be determined experimentally and by theoretical modelling methods (Chatris *et al.*, 2001:1376). According to Chatris *et al.*, (2001:1376) once ignited the fire goes through stages of development, stationary and then deterioration till it is self-extinguished. The time the gel took from ignition till it burned to completion, was used to calculate the burning rate as shown in equation 4.4. The results are presented in Figure 4.6.

$$\text{Burning rate} = \frac{\text{gel burned}(g)}{\text{burning time}(min)} \quad (4.4)$$

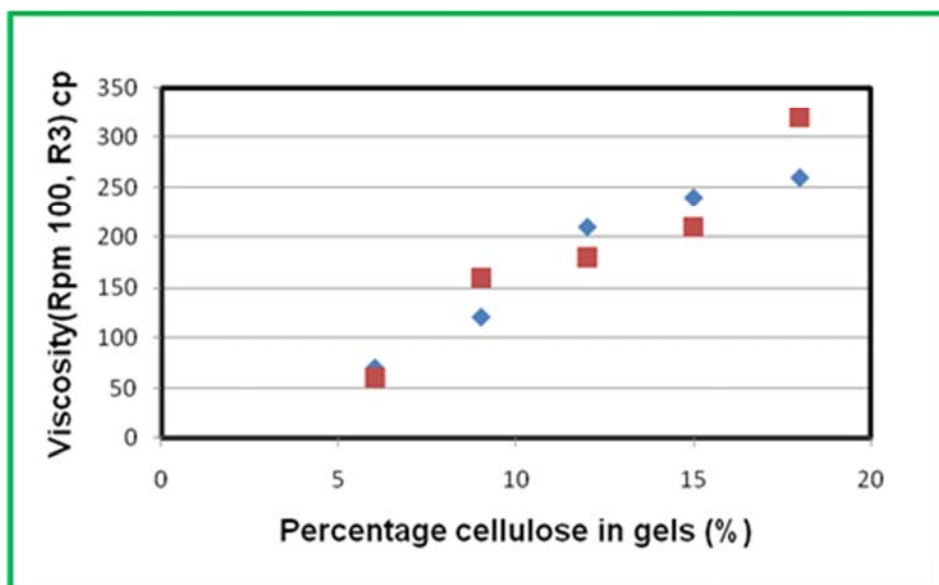


**Figure 4.6** The influence of gelling agent content in the burning rate of prepared gel  
 [■ - *Cereus Jamacaru* gel, ◆ - *Opuntia ficas-indica* gel ]

As the percentage of cellulose in the gel increases the burning rate decreases. A decrease in the burning rate allows for longer burning of the gel. A gel with a larger burning time will be more efficient in providing the necessary heat when used for space heating and cooking than a gel with a short burning time (Mhazo, 2001:2). The burn time of Opuntia 18 (139s) and Cereus 18 (151s) is the longest amongst the prepared gels (see Appendix A ). In terms of burn time, Opuntia 18 and Cereus 18 demonstrated to be better suited for space heating. None of the commercial gels matches the above-stated gels in terms of the burning time as illustrated in Table 4.6.

#### 4.4.3 Viscosity

Viscosity depends on the concentration or molecular weight of a substance at a particular temperature (Tog̃rul & Arslan, 2003: 63) or it can be defined as a measurement of a fluid’s resistance to flow ( Wagner *et al.*, 2010:721). The results of the viscosity of the prepared gels are presented in Figure 4.7.



**Figure 4.7** Effect of gelling agent content on viscosity of the prepared gels

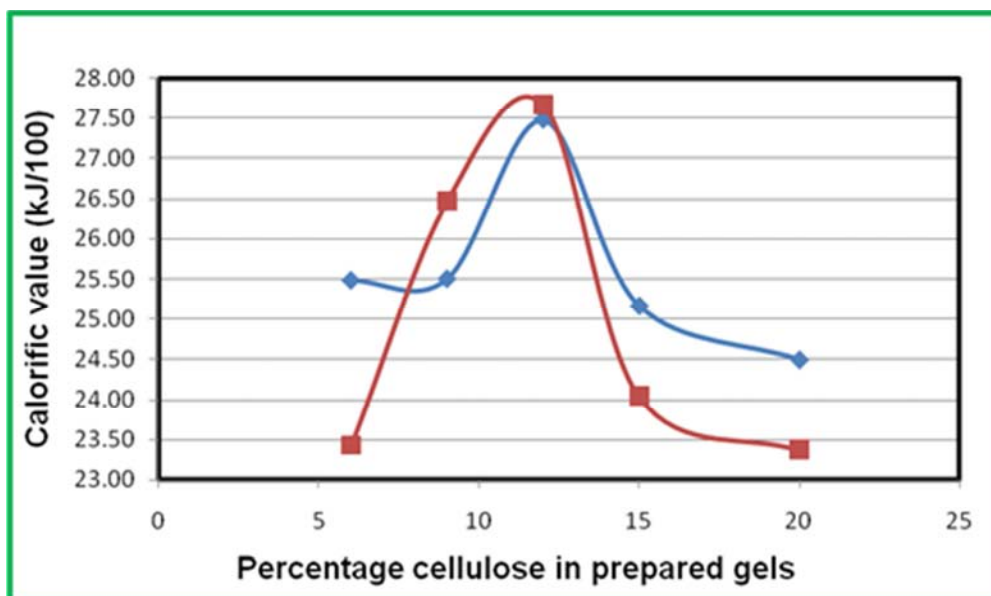
■ - *Cereus Jamacaru* gel, ◆ - *Opuntia ficas-indica*

The gel fuel has to flow easily for better utilisation and therefore viscosity is critical (Wagner *et al.*, 2010:711). The viscosity of prepared gels increases as the gelling agent

content increases (see Figure 4.7). *Opuntia* 18 and *Cereus* 18 have viscosities of 260 cp and 320 cp respectively. According to Table 4.6 the viscosity of commercial gels is higher (i.e. 10670 cp for Green gel, 19020 cp for Blue gel and 15370 cp for Red). The low viscosity of the prepared gels compared to commercial gels, renders them desirable for better utilisation (Wagner *et al.*, 2010:711).

#### 4.4.4 Calorific values

The quantity of heat produced by the combustion of ethanol gel at a constant pressure and temperature is known as the calorific value of the gel. During the combustion process water vapour is released. The amount of heat contained in this water vapour together with the heat produced by the combusted material is referred to as the Gross Calorific Value (GCV) or Higher Heating Value (HHV). In the case where heat from the water vapour is not counted, the quantity of heat produced is referred to as the lower calorific value (LCV) or net heating value (NHV). The results presented in Figure 4.8 reveals the Gross Calorific Values of the prepared gels.



**Figure 4.8** Effect of gelling agent content on the calorific value of prepared gels  
 [■ - *Cereus Jamacaru* gel, ◆ - *Opuntia ficas-indica*]

*Opuntia* 12 and *Cereus* 12 had the highest calorific values at 27.48 kJ/kg and 27.67 kJ/kg respectively. Compared to commercial gels viz. Green gel (14.88 kJ/kg), Blue gel (24.07 kJ/kg) and Red gel (10.81 kJ/kg), *Opuntia* 12 and *Cereus* 12 provide a better heating value per gram of the gel.

### 4.5 Summary

In this investigation the gelling agents (cellulose) from the local cacti, *Opuntia ficus-indica* and *Cereus Jamacaru* were extracted. The experimental yield of *Opuntia ficus-indica* stem (15 wt%  $\pm$  6.7 wt %), *Opuntia ficus-indica* leaves (12.7 wt%  $\pm$  6.8 wt %) and *Cereus Jamacaru* (11.5 wt%  $\pm$  7.8 wt %) show a considerable difference. According to Melainine and co-workers (2003:79) the untreated fibre contains 21.6 wt% cellulose. This correlates with the cellulose content of untreated fibre of *Opuntia* stem (26.23 wt %). The cellulose content of untreated *Cereus Jamacaru* fibre is 21.85 wt%. Even though there is no reported composition of *Cereus Jamacaru* this value suggests that the theoretical yield of *Cereus Jamacaru* and that of *Opuntia* stem show marked differences.

In order to synthesise the ethanol gel, the purity of cellulose was determined by Fourier Transform infrared (FTIR) spectroscopy. After Nitric acid digestion most lignin and hemicelluloses were removed as shown by the absence of the Peak at 1507 cm<sup>-1</sup> (7) (Alemdar & Sain, 2008: 1668) and a small band associated with (C=O) stretching unconjugated ketones at 1718 cm<sup>-1</sup> (6)(Sun *et al.*, 2004:1717 ; Yan Yan *et al.*, 2009:61; Freire *et al.*, 2005:454).

The increase in the intensity of the O–H vibration band at 3200-3500 cm<sup>-1</sup> (1) ( Adel *et al.*, 2010:4449) indicates the exposed O–H functional groups after removal of hemicellulose and lignin. There is an increase in the intensity of C–O stretching and C–H rock vibrations associated with cellulose at 1017 cm<sup>-1</sup> (3) and 891 cm<sup>-1</sup> (5) respectively. As extraction steps from water to nitric acid digestion progresses, the cellulose content follows the increasing trend as reported by Melainine and co-workers (1003:79).

The extracted cellulose of *Opuntia ficas-indica* has less Hemicellulose and lignin than that of *Cereus Jamacaru* (Table 4.3). This is confirmed by the presence of small shoulders of peaks at  $1718\text{ cm}^{-1}$  (6) and  $1507\text{ cm}^{-1}$  (7) associated with Hemicellulose and lignin (Chen *et al.*, 2011:1807) in the spectra of *Cereus Jamacaru* (Figure 4.5) and the absence of the same peaks in the spectra of *Opuntia ficas-indica* (Figure 4.3).

The synthesised ethanol was characterised in order to compare these gels to the commercial gels. The properties determined were burn time, viscosity and calorific value.

### 4.5.1 Burn time

Based on the percentage content of cellulose, the best formulation was determined. *Opuntia* 15 (4.375 wt% cellulose) gave a burn time of  $147\text{s} \pm 7.8\text{s}$  and *Cereus* 18 (5.25 wt% cellulose) gave a burn time of  $151\text{s} \pm 21.3\text{s}$ . The best commercial gel (Blue gel) in terms of burn time gave ( $113\text{s} \pm 1.0\text{s}$ ) which is less than *Opuntia* 15 and *Cereus* 18.

### 4.5.2 Residue

The highest residues of the prepared gels are still lower than those of commercial gels (i.e. Green gel (6.09 %) Blue gel (9.49%))(Table 4.4).The *Cereus* gel residue of 4.2%, which is the highest of prepared gels, is less than 5% as prescribed by the South African Bureau of Standards (SANS 448.,2010:5).

### 4.5.3 Viscosity

The synthesised gels with the highest viscosity viz. *Opuntia* 18 (5.25wt% cellulose) and *Cereus* 18(5.25 wt% cellulose) have viscosities of 260cp and 320cp respectively. The viscosities of these formulations are lower than those of commercial gels, viz. Green gel (10670cp), Blue gel (19020 cp) and Red gel (15370cp). In terms of fluidity *Opuntia* 18 and *Cereus* 18 have a better flow than commercial gels.

### 4.5.4 Calorific value

Gels have a unique way of burning, which is different from that of liquid fuels (Mishra *et al.*, 2011:1805). In the process of combustion the heated fluid creates small bubbles as it struggles to escape from the polymer network. According to Mishra and co-workers (2011:805), the base fuel utilises diffusion as transport phenomenon facilitated by bubble ruptures and micro explosions. This unique way of burning makes alcohols better and cleaner burning fuels (Shaw *et al.*, 2002:30). *Opuntia* 12 (3.5 wt. % cellulose) and *Cereus* 12 (3.5 wt. % cellulose) produce the highest calorific values at 27.48 kJ/Kg and 27.67 kJ/Kg respectively. These values are far better than those of other commercial gels and slightly better than Blue gel which produces a heating value of 24.07 kJ/Kg.

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## CHAPTER 5 - CONCLUSION AND RECOMMENDATION

“We don’t see things as they are, we see them as we are” Anaïs Nin“

“The highest possible stage in moral culture is when we recognize that we ought to control our thoughts.” Charles Darwin

### OVERVIEW

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This chapter is divided into two sections. The conclusions arrived at from the results in this study are presented in Section 5.1 and the recommendations for future experimental work are suggested in Section 5.2.

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#### 5.1 Conclusions

The study was undertaken to meet the energy challenge faced by the rural poor. An attempt was made to produce an alternative energy source to paraffin. The energy source that would be cheaper, renewable, nontoxic, emission free and proudly South African was envisaged. The following conclusions were made, based on the objectives and the results obtained:

##### 5.1.1 Extraction of gelling agents.

Cellulose was successfully extracted from *Cereus Jamacaru* and *Opuntia ficus-indica* by using a method by Malainine *et al.* (2003:78). The purity of extracted cellulose was inferred by the absence of peaks at  $1507\text{ cm}^{-1}$  and  $1718\text{ cm}^{-1}$  associated with lignin (Chen *et al.*, 2011:1807; Sun *et al.*, 2004:1717) and the increase in intensity of O–H vibration band at  $3200\text{-}3500\text{ cm}^{-1}$  associated with cellulose (Sun *et al.*, 2007:2388) using FTIR spectroscopy. The cellulose of *Opuntia ficas-indica* has less Hemicellulose and lignin than that of *Cereus Jamacaru*. This suggests that the extraction protocol followed was effective for *Opuntia ficas-indica*.

The yield of the cellulose was obtained from the *Opuntia ficas-indica* stem (15 wt%  $\pm$  6.7 wt%), *Opuntia ficas-indica* leaves (12.7 wt%  $\pm$  6.8 wt%) and *Cereus Jamacaru* (11.5wt%  $\pm$  7.8 wt%) and indicated that *Opuntia ficus-indica* gave a better yield of cellulose.

### 5.1.2 Characterisation of ethanol gel

Based on the properties of ethanol gel (burn time, residue, viscosity and calorific values), the formulation of the prepared gel that showed better qualities than the commercial gels were *Opuntia* 12 (Calorific value- 26.48 KJ/100g), *Cereus* 12 (Calorific value- 27.67 KJ/100g), *Opuntia* 15 (Burn time-147s  $\pm$  7.8s), *Cereus* 18 ( Burn time-151s  $\pm$  21.3s.), *Opuntia* 18 (Viscosity-260cp ) and *Cereus* 18( Viscosity-320cp).

### 5.2 Recommendation

The results obtained in this study open the way for further development and optimisation of ethanol gel synthesis. Recommendations for future research based on the findings of this work would include the following areas:

- The cellulose content of plants undergo seasonal variations (Black, 1950:386) which is influenced by the species, its age or growth, the locality where it grows and the conditions under which it grows (Black, 1950:386). It is recommended that the effect of age and locality on the cellulose content of *Opuntia ficas-indica* and *Cereus Jamacaru* be investigated.
- According to Hakansson & Ahlgren (2005:182) the viscosity of extracted cellulose depends on the condition of hydrolysis. In this study Nitric acid of concentration 0.05N at 60°C was used in the hydrolysis step. It is recommended that different hydrolyses conditions be explored in order to maximise the viscosity of the extracted cellulose.
- It is recommended that the effect of purity of cellulose on the properties of ethanol gel be researched.

- It is recommended that the composition of gels as prepared according to Table 3.3 be further varied and their properties (i.e. Burn time, Residue, Viscosity and Calorific Values) studied in order to arrive at a formulation that is of commercial viability.

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## APPENDIX A - CALCULATIONS

*“What you cannot measure, you cannot control; what you cannot control, you cannot improve.” D.Beecroft (1992)*

### OVERVIEW

Appendix A presents the calculations that were used to process the experimental data that were obtained. Section A.1 illustrates the calculation of the cellulose yield, whereas Section A.2 describes the calculation of the experimental error. Section A.3 presents the composition of cacti species and Section A.4 shows calculations of gel composition. Experimental errors of Residue and Burn Time are presented in Section A.5

#### A.1 Calculation of Cellulose Yield

The cellulose yield was calculated by Equation A.1:

$$\text{Cellulose yield (wt \%)} = \frac{m_{\text{cellulose}}}{m_{\text{pulp of cacti}}} \dots\dots\dots (\text{A.1})$$

$m_{\text{cellulose}}$  is the mass of cellulose obtained in the final step of extraction and  $m_{\text{pulp of cacti}}$  is the pulp of cacti used as starting sample for the extraction.

#### A.2 Experimental error for the extraction of cellulose

The experimental error of results presented in Section 4.2 was calculated by Equations that follows.

- Step 1 The mean of the data set:

$$\bar{x} = \frac{1}{N} \sum_{i=1}^N x_i \dots\dots\dots (\text{A.2})$$

Where N is the total number of data points and  $x_i$  is an individual data point.

- Step 2 The standard deviation (STDEV) of the data set.

$$\text{STDEV} = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N-1}} \dots\dots\dots (\text{A.3})$$

- Step 3 The confidence interval at 95%.

$$Confidence\ level = \frac{1.96 * STDEV}{\sqrt{N}} \dots\dots\dots(A.4)$$

- Step 4 Percentage error.

$$\% \text{ Error} = \frac{Confidence\ level}{Average} * 100 \dots\dots\dots(A.5)$$

The yield of the cacti was calculated as stipulated in Section 4.1 after determining the moisture using a HR83 Halogen Moisture Analyzer illustrated in Figure 3.8. Percentage error and the yield of *Opuntia ficas-indica* stem, *Opuntia ficas-indica* leaves and *Cereus Jamacara* stem are presented in Table A.1 to Table A.3.

**Table A.1** Experimental error and the yield of *Opuntia ficas-indica* stem

	Weight 1	Weight 2	Weight 3	Average	Standard deviation	Confidence level	% Error
<b>Water extraction</b>							
Starting material(g)	600.04	600.01	600.01	600.02	0.00	0.00	0.00
Residue	452.60	423.81	463.40	446.60	20.50	23.20	5.20
<b>Alkaline extraction</b>							
starting material(g)	452.60	423.81	463.40	446.60	20.50	23.20	5.20
Residue	411.65	399.96	432.61	414.74	16.50	18.70	4.50
<b>Bleach</b>							
starting material(g)	411.65	399.96	432.61	414.74	16.50	18.70	4.50
Residue	376.43	361.50	411.32	383.08	25.60	28.90	7.60
<b>Nitric digestion</b>							
starting material(g)	376.43	361.50	411.32	383.08	25.60	28.90	7.60
Residue	342.22	356.83	289.95	329.67	35.20	39.80	12.10
% Moisture	73.09	73.70	71.09	72.62	1.40	1.50	2.10
Yield (g)	92.14	93.85	83.83	90.26	5.40	6.10	6.70
<b>% yield</b>	<b>15.40</b>	<b>15.60</b>	<b>14.00</b>	<b>15.00</b>	<b>0.90</b>	<b>1.00</b>	<b>6.70</b>

**Table A.2** Experimental error and the yield of *Opuntia ficas-indica* leaves

	<b>Weight 1</b>	<b>Weight 2</b>	<b>Weight 3</b>	<b>Average</b>	<b>Standard deviation</b>	<b>Confidence level</b>	<b>% Error</b>
<b>Water extraction</b>							
Starting material	600.04	600.01	600.01	600.02	0.00	0.00	0.00
Residue	452.06	600.02	600.01	600.02	0.00	0.00	0.00
<b>Alkaline extraction</b>							
starting material(g)	452.06	450.50	468.61	457.06	10.00	11.40	2.50
Residue	412.50	450.05	399.98	414.84	16.20	18.30	4.40
<b>Bleach</b>							
Starting material(g)	412.50	450.05	399.98	414.84	16.20	18.30	4.40
Residue	378.90	346.95	339.63	355.16	20.90	23.60	6.70
<b>Nitric digestion</b>							
Starting material(g)	378.90	346.95	339.63	355.16	20.90	23.60	6.70
Residue	342.56	309.58	295.83	315.99	24.00	27.20	8.60
% Moisture	81.00	76.65	77.90	78.52	2.20	2.50	3.20
Yield (g)	71.99	80.99	75.06	76.02	4.60	5.20	6.80
<b>% yield</b>	<b>12.00</b>	<b>13.5</b>	<b>12.5</b>	<b>12.70</b>	<b>0.80</b>	<b>0.90</b>	<b>6.80</b>

**Table A.3** Experimental error and the yield of *Cereus Jamacaru* stem

	<b>Weight 1</b>	<b>Weight 2</b>	<b>Weight 3</b>	<b>Average</b>	<b>STDEV</b>	<b>Confidence level</b>	<b>% Error</b>
<b>Water extraction</b>							
Starting material(g)	600.04	600.01	600.01	600.02	0.00	0.00	0.00
Residue	449.08	452.05	461.56	454.23	6.50	7.40	1.60
<b>Alkaline extraction</b>							
starting material(g)	449.08	452.05	461.56	454.23	6.50	7.40	1.60
Residue	345.05	336.36	341.41	340.94	4.40	4.90	1.40
<b>Bleach</b>							
starting material(g)	345.05	336.36	341.41	340.94	4.40	4.90	1.40
Residue	314.40	289.05	335.54	312.99	23.3	26.30	8.40
<b>Nitric digestion</b>							
starting material(g)	314.40	289.05	335.54	312.99	23.30	26.30	8.40
Residue	244.06	246.03	240.04	243.38	3.10	3.50	1.40
% Moisture	79.33	76.65	77.90	77.96	1.30	1.50	1.90
Yield (g)	64.98	67.48	74.15	68.98	4.70	5.40	7.80
<b>% yield</b>	<b>10.80</b>	<b>11.20</b>	<b>12.4</b>	<b>11.50</b>	<b>0.80</b>	<b>0.90</b>	<b>7.80</b>

### **A.3 Composition of *Cereus Jamacaru* and *Opuntia ficas-indica***

Components of the alkaline insoluble residue (Cellulose, Hemicellulose and Lignin) for *Cereus Jamacaru* and *Opuntia ficas-indica* were calculated as explained in Section 4.3.1. The results are presented in Table A.4.

**Table A.4** Calculation of the composition of cacti

Sample	NDF(%)	ADF(%)	ADL(%)	Cellulose	hemicellulose	lignin
				ADF-ADL	NDF-ADF	ADL
<b>Cereus</b>						
Raw	46.16	25.54	3.69	21.85	20.62	3.69
Water extraction	50.59	29.43	3.47	25.96	21.16	3.47
Bleach	44.32	30.12	3.92	26.20	14.20	3.92
Alkaline extraction	42.33	31.09	4.15	26.94	11.24	4.15
Nitric extraction	50.3	43.16	3.38	39.78	7.14	3.38
<b>Opuntia leaves</b>						
Bleach	44.62	42.23	4.22	38.01	2.39	4.22
Nitric extraction	53.93	52.43	6.53	45.90	1.50	6.53
<b>Opuntia stem</b>						
Raw	49.15	29.1	2.87	26.23	20.05	2.87
Water extraction	51.07	24.34	2.41	21.93	26.73	2.41
<b>Bleach</b>	36.54	31.43	5.06	26.37	5.11	5.06
Alkaline extraction	36.96	35.32	4.48	30.84	1.64	4.48
Nitric extraction	60.02	59.27	2.57	56.70	0.75	2.57

#### A.4 Calculation of components of gels

Five gels of each cactus were prepared as explained in Section 3.4. Cellulose of 65 % moisture was used in all the preparations. Ethanol was maintained above 80% in all prepared gels. The moisture content of cellulose was accounted as part of the water component of the gel as calculated in Table A.5 for *Opuntia ficas-indica* and Table A.6 for *Cereus Jamacaru*.

**Table A.5** Ethanol gel compositions of *Opuntia ficas-indica*

gel	Cellulose (g) (Wet basis)	Cellulose (Dry basis)	Cellulose (%)	Moisture (g)	Moisture (g)	Ethanol (g)	Ethanol (%)	Tot. wt (g)
6	6.00	2.12	1.75	3.90	3.25	114	95	120
9	9.00	3.15	2.63	5.85	4.88	111	93	120
12	12.00	4.20	3.50	7.80	6.50	108	90	120
15	15.00	5.25	4.38	9.75	8.13	105	88	120
18	18.00	6.30	5.25	11.70	9.75	102	85	120

**Table A.6** Ethanol gel compositions of *Cereus Jamacaru*

gel	Cellulose (g) (Wet basis)	Cellulose (Dry basis)	Cellulose (%)	Moisture (g)	Moisture (g)	Ethanol (g)	Ethanol (%)	Tot. wt (g)
6	6	2.10	1.75	3.90	3.25	114	95	120
9	9	3.15	2.63	5.85	4.88	111	93	120
12	12	4.20	3.50	7.80	6.50	108	90	120
15	15	5.25	4.38	9.75	8.13	105	88	120
18	18	6.30	5.25	11.70	9.75	102	85	120

**A.5 Calculation of experimental error of Residue and Burn Time of gels.**

The results of the residue and burn time of each gel are presented in Tables A.7 to A.10.

**Table A.7** Experimental error of residue and burn time of *Opuntia 6*

<b>Opuntia 6</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin.</b>		<b>Gel</b>		<b>Burn time (s)</b>	<b>gel/time (g/min)</b>
			<b>mass (g)</b>	<b>Residue (g)</b>	<b>burned (g)</b>	<b>Residue (%)</b>		
Run 1	47.17	5.05	47.19	0.02	5.03	0.33	85.00	3.55
Run2	46.90	5.07	46.91	0.02	5.05	0.32	79.00	3.84
Run 3	47.14	5.09	47.16	0.01	5.07	0.29	87.00	3.50
Average		5.07		0.02	5.05	0.31	83.67	3.62
Std deviation	0.15	0.02	0.15	0.00	0.02	0.02	4.16	0.31
Confidence		0.02		0.00	0.02	0.02	4.71	0.31
% Error		0.46		6.56	0.48	6.99	5.63	5.09

**Table A.8** Experimental error of residue and burn time *Opuntia 9*

<b>Opuntia 9</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin.</b>		<b>Gel</b>		<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
			<b>Mass (g)</b>	<b>Residue (g)</b>	<b>burned (g)</b>	<b>Residue (%)</b>		
Run 1	46.43	5.06	46.52	0.09	4.97	1.76	84.00	3.55
Run 2	46.77	5.01	46.85	0.08	4.93	1.57	87.00	3.40
Run 3	47.87	5.07	47.94	0.07	5.00	1.36	88.00	3.41
Average		5.04		0.08	4.97	1.56	86.33	3.45
Std. deviation		0.03		0.01	0.04	0.20	2.08	1.02
Confidence		0.04		0.01	0.04	0.23	2.36	1.02
% Error		0.75		14.49	0.81	14.51	2.73	17.75

**Table A.9** Experimental error of residue and burn time *Opuntia 12*

<b>Opuntia 12</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.85	5.10	46.91	0.05	5.04	1.07	82.00	3.69
Run 2	47.04	5.09	47.14	0.09	5.00	1.87	91.00	3.30
Run 3	46.60	5.08	46.69	0.09	4.99	1.82	95.00	3.15
Average		5.09		0.08	5.01	1.58	89.33	3.37
Std. deviation		0.01		0.02	0.03	0.45	6.66	0.25
Confidence		0.01		0.03	0.03	0.51	7.53	0.25
% Error		0.15		32.07	0.63	32.16	8.43	4.49

**Table A.10** Experimental error of residue and burn time *Opuntia 15*

<b>Opuntia 15 Unit</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.88	5.99	46.90	0.02	5.96	0.4010	134.00	2.679
Run 2	47.55	5.87	47.59	0.04	5.845	0.5981	138.00	2.54
Run 3	46.66	5.99	46.69	0.04	5.96	0.5784	137.00	2.61
Average		5.95		0.03	5.92	0.53	136.33	2.61
Std. deviation		0.10		0.00	0.10	0.1	2.10	2.12
Confidence		0.10		0.00	0.10	0.1	2.40	2.12
% Error		1.30		22.70	1.40	23.4	1.70	48.77
Average		5.95		0.03	5.92	0.526	136.33	2.61

**Table A.11** Experimental error of residue and burn time of *Opuntia 18*

<i>Opuntia 18</i>	M of tin (g)	M of gel (g)	Fin. Mass (g)	Residue (g)	Gel		Burn time (s)	Gel/Burn
					burned (g)	Residue (%)		time (g/min)
Run 1	46.83	5.07	46.98	0.15	4.92	3.00	138.00	2.14
Run 2	46.55	5.05	46.70	0.16	4.90	3.07	151.00	1.95
Run 3	46.80	5.01	46.96	0.16	4.86	3.16	128.00	2.28
Average		5.05		0.16	4.89	3.08	139.00	2.11
STDEV		0.03		0.00	0.03	0.08	11.53	0.17
Confidence		0.03		0.00	0.04	0.09	13.05	0.17
% Error		0.69		2.16	0.78	2.85	9.39	4.96

**Table A.12** Experimental error of residue and burn time of *Cereus 6*

<b>Cereus 6</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.84	5.05	46.87	0.03	5.02	0.60	86.00	3.50
Run 2	46.04	5.06	46.05	0.01	5.04	0.25	88.00	3.44
Run 3	46.75	5.04	46.79	0.03	5.01	0.66	92.00	3.27
Average		5.05		0.03	5.02	0.50	88.67	3.40
Std. deviation		0.01		0.01	0.02	0.22	3.06	0.33
Confidence		0.01		0.01	0.02	0.25	3.46	0.33
% Error		0.00		0.50	0.00	0.50	0.04	5.78

**Table A.13** Experimental error of residue and burn time of *Cereus 9*

<b>Cereus 9</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.90	5.05	46.99	0.09	4.97	1.69	89.00	3.35
Run 2	46.83	5.05	46.90	0.07	4.98	1.44	92.00	3.25
Run 3	47.52	5.02	47.60	0.08	4.95	1.50	88.00	3.37
Average		5.04		0.08	4.97	1.54	89.67	3.32
STDEV		0.02		0.01	0.02	0.13	2.08	0.48
Confidence		0.02		0.01	0.02	0.15	2.36	0.48
% Error		0.40		9.81	0.38	9.66	2.63	8.64

**Table A.14** Experimental error of residue and burn time of *Cereus 12*

<b>Cereus 12</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.32	5.01	46.47	0.15	4.86	3.08	85.00	3.43
Run 2	47.01	5.04	47.16	0.16	4.88	3.09	90.00	3.25
Run 3	46.43	5.02	46.58	0.15	4.87	3.04	87.00	3.36
Average		5.02		0.15	4.87	3.07	87.33	3.35
Std. deviation		0.01		0.00	0.01	0.03	2.52	0.24
Confidence		0.01		0.00	0.01	0.03	2.85	0.24
% Error		0.24		1.23	0.23	1.13	3.26	4.24

**Table A.15** Experimental error of residue and burn time of *Cereus 15*

<b>Cereus 15</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	47.71	4.14	47.90	0.19	3.96	4.76	82.00	2.89
Run 2	47.73	4.17	47.90	0.17	4.00	4.27	84.00	2.86
Run 3	47.88	4.00	48.00	0.12	3.88	3.01	83.00	2.81
Average	47.77	4.11	47.93	0.16	3.95	4.02	83.00	2.85
Std. deviation	0.09	0.09	0.06	0.04	0.06	0.90	1.00	0.04
Confidence	0.10	0.10	0.06	0.04	0.07	1.02	1.13	0.05
% Error	0.22	2.52	0.13	26.54	1.68	25.45	1.36	1.74

**Table A.16** Experimental error of residue and burn time of *Cereus 18*

<b>Cereus 18</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	46.6551	5.0928	46.8606	0.2055	4.8873	4.0351	184	1.5937
Run 2	46.8849	5.0929	47.1011	0.2162	4.8767	4.2451	138	2.1203
Run 3	46.2135	5.0463	46.4311	0.2176	4.8287	4.3121	132	2.1949
Average		5.0773		0.2131	4.8642	4.1971	151.33	1.9286
Std deviation		0.0		0.0	0.0	0.1	28.45	0.0659
Confidence		0.0		0.0	0.0	0.2	32.19	0.0659
% Error		0.6		3.5	0.7	3.9	21.27	2.0489

**Table A.17** Experimental error of residue and burn time of Green gel

<b>Green gel</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	47.35	5.02	47.65	0.30	4.72	6.02	109.00	2.60
Run 2	47.02	5.09	47.34	0.31	4.77	6.18	110.00	2.60
Run 3	46.46	5.03	46.77	0.31	4.73	6.07	107.00	2.65
Average		5.05		0.31	4.74	6.09	108.67	2.62
Std. deviation		0.04		0.01	0.03	0.08	1.53	1.13
Confidence		0.04		0.01	0.03	0.09	1.73	1.13
% Error		0.79		2.31	0.69	1.53	1.59	26.01

**Table A.18** Experimental error of residue and burn time of Blue gel

<b>Blue gel</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	47.34	5.04	47.82	0.48	4.56	9.52	113.00	2.42
Run 2	47.02	5.01	47.50	0.48	4.54	9.48	112.00	2.43
Run 3	47.52	5.01	47.99	0.47	4.54	9.46	114.00	2.39
Average		5.02		0.48	4.55	9.49	113.00	2.41
Std. deviation		0.02		0.00	0.01	0.03	1.00	0.84
Confidence		0.02		0.00	0.02	0.03	1.13	0.84
% Error		0.38		0.69	0.35	0.32	1.00	20.89

**Table A.19** Experimental error of residue and burn time of Red gel

<b>Red gel</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	46.46	5.08	46.47	0.01	5.07	0.21	91.00	3.34
Run 2	46.56	5.08	46.58	0.01	5.07	0.21	94.00	3.24
Run 3	46.21	5.05	46.22	0.01	5.04	0.21	93.00	3.25
Average		5.07		0.01	5.06	0.21	92.67	3.28
Std. deviation		0.02		0.00	0.02	0.00	1.53	0.68
Confidence		0.02		0.00	0.02	0.00	1.73	0.68
% Error		0.39		1.06	0.39	0.75	1.87	12.52

**Table A.20** Viscosity and cellulose content of prepared gels of *Opuntia ficas-indica*

<b>Gel type</b>	<b>% Cellulose</b>	<b>% Moisture</b>	<b>% ethanol</b>	<b>Viscosity(Rpm 100, R3) cp</b>
<i>Opuntia 6</i>	1.75	3.25	95.00	70.00
<i>Opuntia 9</i>	2.63	4.88	92.50	120.00
<i>Opuntia 12</i>	3.50	6.50	90.00	210.00
<i>Opuntia 15</i>	4.38	8.13	87.50	240.00
<i>Opuntia 18</i>	5.25	9.75	85.00	260.00

**Table A.21** Viscosity and cellulose content of prepared gels of *Cereus Jamacaru*

<b>Gel type</b>	<b>% Cellulose</b>	<b>% Moisture</b>	<b>% ethanol</b>	<b>Viscosity(Rpm 100, R3) cp</b>
<i>Cereus 6</i>	1.75	3.25	95.00	60.00
<i>Cereus 9</i>	2.63	4.88	92.50	160.00
<i>Cereus 12</i>	3.50	6.50	90.00	180.00
<i>Cereus 15</i>	4.38	8.13	87.50	210.00
<i>Cereus 18</i>	5.25	9.75	85.00	320.00

**Table A.22** Properties of commercial gels

	<b>Green gel</b>	<b>Blue gel</b>	<b>Red gel</b>	<b>Paraffin</b>
Burn time (s)	108.67	113.00	92.67	86.67
Gel time (g/min)	2.62	2.41	3.28	5.73
Viscosity (100 Rpm, R3) cp	10670.00	19020.00	15370.00	X
Ash content (Residue (%))	6.09	9.49	0.21	1.65
Caloric values (kJ/kg)	14.88	24.07	10.81	44.70 <sup>a</sup>

[X- not given]

**Table A. 23** Calorific values of Prepared gels<sup>a</sup>

	<b>Calorific values of prepared gels (kJ/kg)</b>				
	<b>6</b>	<b>9</b>	<b>12</b>	<b>15</b>	<b>18</b>
Cellulose content					
<i>Opuntia ficas-indica</i>	25.48	25.5	27.48	25.16	24.49
<i>Cereus Jamacaru</i>	23.43	26.49	27.67	24.04	23.37

[a - Results provided by Agricultural Research Council (ARC)]

## APPENDIX B - EXPERIMENTAL DATA

*“Blessed is he who has learned to admire but not envy, to follow but not imitate, to praise but not flatter, and to lead but not manipulate.”* W.A Ward

*“It is the province of knowledge to speak; it is the privilege of wisdom to listen.”* Christine Lane.

### OVERVIEW

The data obtained from the study are listed in Appendix B. Section B.1 presents the yield of cellulose. Section B.2 presents the residue and burn time of gels. Viscosity data are shown in Section B.3, with calorific values and composition data in Sections B.4 and B.5 respectively.

#### B.1 Cellulose extraction data

Tables B.1 to B.3 list the yields obtained from *Opuntia ficas-indica* stem, *Opuntia ficas-indica* leaves and *Cereus Jamacara* stem respectively.

**Table B.1** Extraction data of *Opuntia ficas-indica* stem.

		Weight 1	Weight 2	Weight 3	Average
Water extraction	Starting material(g)	600.04	600.01	600.01	600.02
	Residue	452.60	423.81	463.40	446.60
Alkaline extraction	starting material(g)	452.60	423.81	463.40	446.60
	Residue	411.65	399.96	432.61	414.74
Bleach	starting material(g)	411.65	399.96	432.61	414.74
	Residue	376.43	361.50	411.32	383.08
Nitric acid digestion	starting material(g)	376.43	361.50	411.32	383.08
	Residue	342.223	356.83	289.95	329.67
	% Moisture	73.08	73.70	71.09	72.62
	Yield (g)	92.14	93.85	83.83	90.26
	% yield	15.40	15.60	14.00	15.00

**Table B.2** Extraction data of *Opuntia ficas-indica* leaves

		Weight 1	Weight 2	Weight 3	Average
Water extraction	Starting material(g)	600.04	600.01	600.01	600.02
	Residue	452.06	450.50	468.61	457.06
Alkaline extraction	starting material(g)	452.06	450.50	468.61	457.06
	Residue	412.50	432.05	399.98	414.84
Bleach	starting material(g)	412.50	450.50	399.98	420.99
	Residue	378.90	346.95	339.63	355.16
Nitric acid digestion	starting material(g)	378.90	346.95	339.63	355.16
	Residue	342.56	309.58	295.83	315.99
	% Moisture	81.00	76.65	77.90	78.52
	Yield (g)	71.99	80.99	75.06	76.02
	% yield	12.00	13.50	12.50	12.70

**Table B.3** Extraction data of *Cereus Jamacaru* stem

		Weight 1	Weight 2	Weight 3	Average
Water extraction	Starting material(g)	600.04	600.01	600.01	600.02
	Residue	452.06	450.50	468.61	457.06
Alkaline extraction	starting material(g)	452.06	450.50	468.61	457.06
	Residue	412.50	432.05	399.98	414.84
Bleach	starting material(g)	412.50	450.50	399.98	420.99
	Residue	378.90	346.95	339.63	355.16
Nitric acid digestion	starting material(g)	378.90	346.95	339.63	355.16
	Residue	342.56	309.58	295.83	315.99
	% Moisture	81.00	76.65	77.90	78.52
	Yield (g)	71.99	80.99	75.06	76.02
	% yield	12.00	13.50	12.50	12.70

## B.2 Composition data

**Table B.4** Chemical composition of *Opuntia ficas-indica* stem, *Opuntia ficas-indica* leaves and *Cereus Jamacara* stem

Sample	NDF(%)	ADF(%)	ADL(%)
<b>Cereus Stem</b>			
Raw	46.16	25.54	3.69
Water extraction	50.59	29.43	3.47
Bleach	44.32	30.12	3.92
Alkaline extraction	42.33	31.09	4.15
Nitric extraction	50.3	43.16	3.38
<b>Opuntia leaves</b>			
Bleach	44.62	42.23	4.22
Nitric extraction	53.93	52.43	6.53
<b>Opuntia stem</b>			
Raw	49.15	29.1	2.87
Water extraction	51.07	24.34	2.41
Bleach	36.54	31.43	5.06
Alkaline extraction	36.96	35.32	4.48
Nitric extraction	60.02	59.27	2.57

[NDF- Neutral Detergent fibre, ADF- Acid Detergent fibre, ADL- Acid Detergent Lignin]

**B.3 Ethanol gel composition data****Table B.5** Ethanol gel compositions of *Opuntia ficas-indica*

Opuntia	Cellulose (g) (Wet basis)	Cellulose (g) dry basis	Cellulose (%)	Moisture (g)	Moisture (%)	Ethanol (g)	Ethanol (%)	Tot. wt (g)
6	6.00	2.12	1.75	3.90	3.25	114	95	120
9	9.00	3.15	2.63	5.85	4.88	111	93	120
12	12.00	4.20	3.50	7.80	6.50	108	90	120
15	15.00	5.25	4.38	9.75	8.13	105	88	120
18	18.00	6.30	5.25	11.70	9.75	102	85	120

**Table B.6** Ethanol gel composition of *Cereus Jamacaru*

Cereus	Cellulose(g) (Wet basis)	Cellulose (g) dry basis	Cellulose (%)	Moisture (g)	Moisture (g)	Ethanol (g)	Ethanol (%)	Tot. wt (g)
6	6	2.10	1.75	3.90	3.25	114	95	120
9	9	3.15	2.63	5.85	4.88	111	93	120
12	12	4.20	3.50	7.80	6.50	108	90	120
15	15	5.25	4.38	9.75	8.13	105	88	120
18	18	6.30	5.25	11.70	9.75	102	85	120

**B.4 Residue and Burn Time data****Table B.7** *Opuntia 6 gel*

<b>Opuntia 6</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>gel/time (g/min)</b>
Run 1	47.17	5.05	47.19	0.02	5.03	0.33	85.00	3.55
Run2	46.90	5.07	46.91	0.02	5.05	0.32	79.00	3.84
Run 3	47.14	5.09	47.16	0.01	5.07	0.29	87.00	3.50
Average		5.07		0.02	5.05	0.31	83.67	3.62

**Table B.8** *Opuntia 9 gel*

<b>Opuntia 9</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.43	5.06	46.52	0.09	4.97	1.76	84.00	3.55
Run 2	46.77	5.01	46.85	0.08	4.93	1.57	87.00	3.40
Run 3	47.87	5.07	47.94	0.07	5.00	1.36	88.00	3.41
Average		5.04		0.08	4.97	1.56	86.33	3.45

**Table B.9** *Opuntia 12 gel*

<b>Opuntia 12</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.85	5.10	46.91	0.05	5.04	1.07	82.00	3.69
Run 2	47.04	5.09	47.14	0.09	5.00	1.87	91.00	3.30
Run 3	46.60	5.08	46.69	0.09	4.99	1.82	95.00	3.15
Average		5.09		0.08	5.01	1.58	89.33	3.37

**Table B.10** *Opuntia 15 gel*

<b>Opuntia 15 Unit</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.88	5.99	46.90	0.02	5.96	0.4010	134.00	2.679
Run 2	47.55	5.87	47.59	0.04	5.845	0.5981	138.00	2.54
Run 3	46.66	5.99	46.69	0.04	5.96	0.5784	137.00	2.61
Average		5.95		0.03	5.92	0.53	136.33	2.61

**Table B.11** *Opuntia 18* gel

<b><i>Opuntia 18</i></b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.83	5.07	46.98	0.15	4.92	3.00	138.00	2.14
Run 2	46.55	5.05	46.70	0.16	4.90	3.07	151.00	1.95
Run 3	46.80	5.01	46.96	0.16	4.86	3.16	128.00	2.28
Average		5.05		0.16	4.89	3.08	139.00	2.11

**Table B.12** *Cereus 6* gel

<b><i>Cereus 6</i></b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.84	5.05	46.87	0.03	5.02	0.60	86.00	3.50
Run 2	46.04	5.06	46.05	0.01	5.04	0.25	88.00	3.44
Run 3	46.75	5.04	46.79	0.03	5.01	0.66	92.00	3.27
Average		5.05		0.03	5.02	0.50	88.67	3.40

**Table B.13** *Cereus 9 gel*

<b>Cereus 9</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.90	5.05	46.99	0.09	4.97	1.69	89.00	3.35
Run 2	46.83	5.05	46.90	0.07	4.98	1.44	92.00	3.25
Run 3	47.52	5.02	47.60	0.08	4.95	1.50	88.00	3.37
Average		5.04		0.08	4.97	1.54	89.67	3.32

**Table B.14** *Cereus 12 gel*

<b>Cereus 12</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. Mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/Burn time (g/min)</b>
Run 1	46.32	5.01	46.47	0.15	4.86	3.08	85.00	3.43
Run 2	47.01	5.04	47.16	0.16	4.88	3.09	90.00	3.25
Run 3	46.43	5.02	46.58	0.15	4.87	3.04	87.00	3.36
Average		5.02		0.15	4.87	3.07	87.33	3.35

**Table B.15** *Cereus 15 gel*

<b>Cereus 15</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	47.71	4.14	47.90	0.19	3.96	4.76	82.00	2.89
Run 2	47.73	4.17	47.90	0.17	4.00	4.27	84.00	2.86
Run 3	47.88	4.00	48.00	0.12	3.88	3.01	83.00	2.81
Average	47.77	4.11	47.93	0.16	3.95	4.02	83.00	2.85

**Table B.16** *Cereus 18 gel*

<b>Cereus 18</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	46.66	5.09	46.86	0.21	4.89	4.04	184.00	1.59
Run 2	46.88	5.09	47.10	0.22	4.88	4.25	138.00	2.12
Run 3	46.21	5.05	46.43	0.22	4.83	4.31	132.00	2.19
Average		5.08		0.21	4.86	4.20	151.33	1.93

**Table B.17** Green gel

<b>Green gel</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	47.35	5.02	47.65	0.30	4.72	6.02	109.00	2.60
Run 2	47.02	5.09	47.34	0.31	4.77	6.18	110.00	2.60
Run 3	46.46	5.03	46.77	0.31	4.73	6.07	107.00	2.65
Average		5.05		0.31	4.74	6.09	108.67	2.62

**Table B.18** Blue gel

<b>Blue gel</b>	<b>M of tin (g)</b>	<b>M of gel (g)</b>	<b>Fin. mass (g)</b>	<b>Residue (g)</b>	<b>Gel burned (g)</b>	<b>Residue (%)</b>	<b>Burn time (s)</b>	<b>Gel/time (g/min)</b>
Run 1	47.34	5.04	47.82	0.48	4.56	9.52	113.00	2.42
Run 2	47.02	5.01	47.50	0.48	4.54	9.48	112.00	2.43
Run 3	47.52	5.01	47.99	0.47	4.54	9.46	114.00	2.39
Average		5.02		0.48	4.55	9.49	113.00	2.41

**Table B.19** Red gel

Red gel	M of tin (g)	M of gel (g)	Fin. mass (g)	Residue (g)	Gel burned (g)	Residue (%)	Burn time (s)	Gel/time (g/min)
Run 1	46.46	5.08	46.47	0.01	5.07	0.21	91.00	3.34
Run 2	46.56	5.08	46.58	0.01	5.07	0.21	94.00	3.24
Run 3	46.21	5.05	46.22	0.01	5.04	0.21	93.00	3.25
Average		5.07		0.01	5.06	0.21	92.67	3.28

**Table B.20** Viscosity and cellulose content of prepared gels of *Opuntia ficas-indica*

Gel type	% Cellulose	% Moisture	% ethanol	Viscosity(Rpm 100, R3) cp
<i>Opuntia 6</i>	1.75	3.25	95.00	70.00
<i>Opuntia 9</i>	2.63	4.88	92.50	120.00
<i>Opuntia 12</i>	3.50	6.50	90.00	210.00
<i>Opuntia 15</i>	4.38	8.13	87.50	240.00
<i>Opuntia 18</i>	5.25	9.75	85.00	260.00

**Table B.21** Viscosity and cellulose content of prepared gels of *Cereus Jamacaru*

<b>Gel type</b>	<b>% Cellulose</b>	<b>% Moisture</b>	<b>% ethanol</b>	<b>Viscosity(Rpm 100, R3) cp</b>
<i>Cereus 6</i>	1.75	3.25	95.00	60.00
<i>Cereus 9</i>	2.63	4.88	92.50	160.00
<i>Cereus 12</i>	3.50	6.50	90.00	180.00
<i>Cereus 15</i>	4.38	8.13	87.50	210.00
<i>Cereus 18</i>	5.25	9.75	85.00	320.00

### **B.5 References**

GEBREMARIAM, T., MELAKU, S. & YAMI, A. 2006. Effects of different levels of cactus (*Opuntia ficus-indica*) inclusion on feed intake, digestibility and body weight gain in tef (*Eragrostis tef*) straw-based feeding of sheep. *Animal Food Science and Technology*, 131: 42-51.

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SOUTH AFRICAN NATIONAL STANDARD.2010. Ethanol gel for cooking and other gel burning appliances: SABS Standards Division: Pretoria. 11p. January. (SANS 448:2010).

TEGEGNE, F., KIJORA, C. & PETERS, K.J. 2005. Effects of incorporating cactus pear (*Opuntia ficus-indica*) and urea-treatment of straw on the performance of sheep. Conference on International Agricultural Research for Development. 1-6.

## APPENDIX C – FT-IR

*“Men of genius are admired. Men of wealth are envied. Men of power are feared, but only men of character are trusted.” Zig Ziglar*

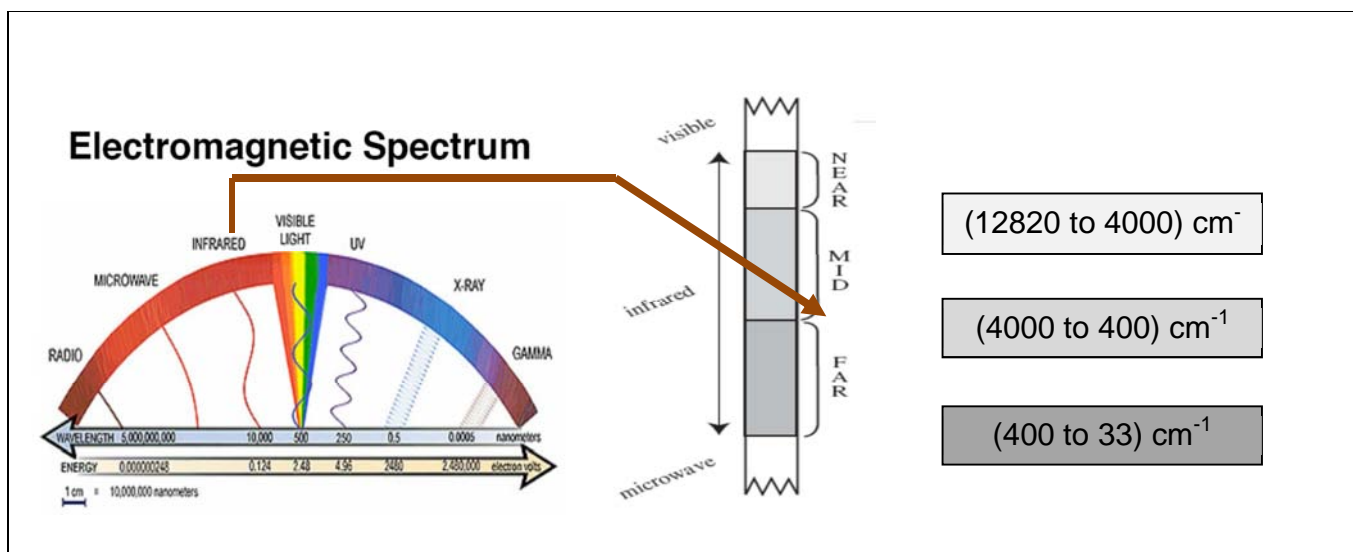
*“We do not inherit the land from our ancestors; we borrow it from our children”* Native American proverb

### OVERVIEW

The background information on FT-IR is presented in Appendix C. Section C.1 presents the Principles of Fourier Transform Infrared. Section C.2 shows the presentation of spectra.

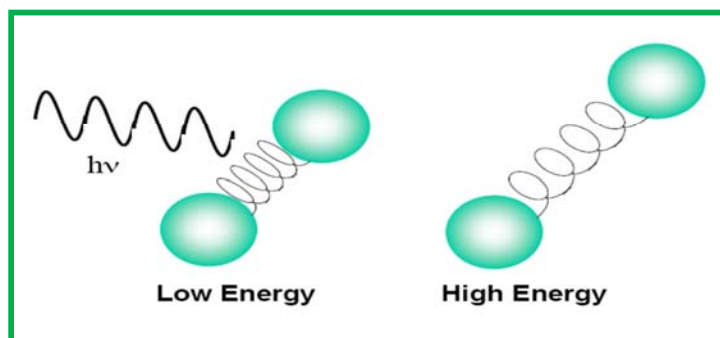
#### C1. Principles of Fourier Transform Infrared (FT-IR)

Infrared Spectroscopy is used both as an identification tool to gather information about the structure of a compound and an analytical tool to assess the purity of a compound. Based on the electromagnetic spectrum, Infrared used in this study is in the range of  $4000\text{--}400\text{ cm}^{-1}$  known as the mid IR range as shown in Figure C.1.



**Figure C.1** The IR regions of the electromagnetic spectrum

Molecules have bond lengths and bond angles which absorb energy as shown in Figure C.2.

**Figure C.2** Absorption of energy by the bond of a molecule

There are two types of vibrations, *i.e.* stretching and bending.

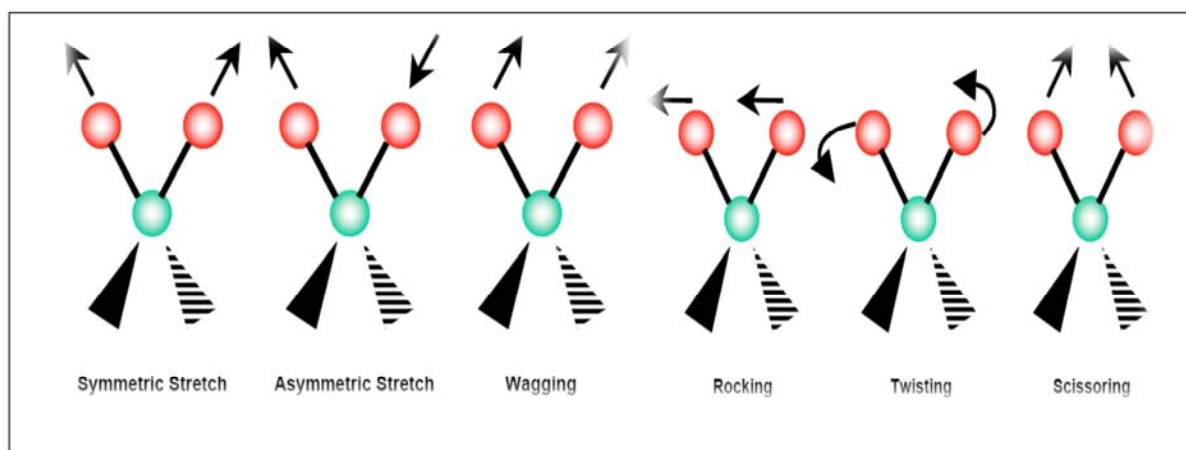
- **Stretching**

Vibration along a bond axis which increases or decreases the bond length (springs)

- **Bending**

Vibration leading to changes in bond angles.

The absorbed energies lead to molecular vibrations that give rise to bands at different frequencies. Molecular vibrations are of many variations as shown in Figure C.4.



**Figure C.3** Different types of vibrations

## C.2 Presentation of spectra

Peaks of IR Spectra provide important information about molecules. Not only the presence of peaks is important, but also absence of peaks that is also of key importance in identifying compounds. The IR Spectra are divided into three regions as illustrated in Figure C.5.

- **Functional group region (4000-1300  $\text{cm}^{-1}$ )**

The functional group region is characterised by fundamental absorption bands of O-H, C=O, C=N Vibrations.

- **Fingerprint region (1300-910  $\text{cm}^{-1}$ )**

This region is unique for each chemical species and not useful for assigning functional groups.

- **Aromatic region (910-650  $\text{cm}^{-1}$ )**

The presence of strong peaks in this region indicates that a compound is aromatic. It is characterised by C-H bending and ring bending bands. The absence of peaks indicates absence of aromatic compound.

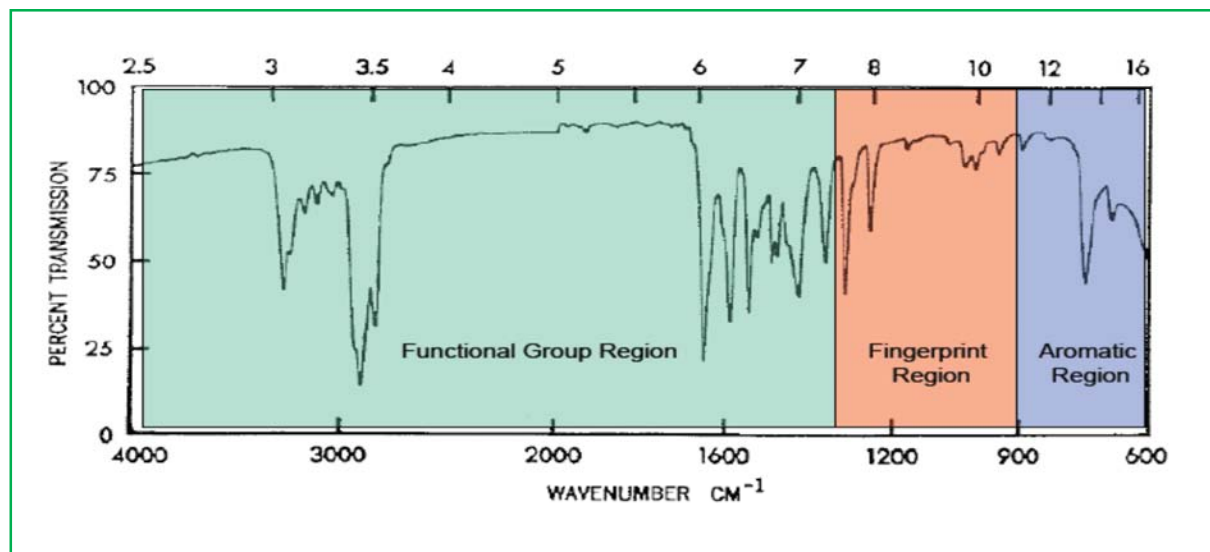


Figure C.4 Three regions of IR spectra

## APPENDIX D – GLOSSARY

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Cellulose	A homopolysaccharide.
<i>Grid</i> –	The generic term used to describe both the National Electricity Grid; being all electricity networks of licensed electricity distributors and transmitters within South Africa, and the Eskom transmission system.
sustainable development	development that meets the needs of the present without compromising the ability of future generations to meet their own needs (John Elkington)
Cellulose–NaOH complexes.	Complexes formed by swelling cellulose I (Native cellulose) with concentrated NaOH. These complexes can recrystallise in an anti-parallel manner to form the energetically favourable cellulose II polymorph after removal of the swelling agent with water (O’Sullivan, 1997)
Hemicellulose	Branched heteropolysaccharides that are structurally homologous to cellulose because they have a backbone composed of 1, 4-linked $\beta$ -D-hexosyl residues. They are found in primary and secondary walls of plants.  Examples: Xyloglucan , Glucoronoxylan, Arabinoxylan, Glucomannan, Galactomannan etc.
Mercerisation	The process involving swelling of native cellulose I fibres in concentrated sodium hydroxide with formation of cellulose– NaOH complexes. Mercerisation of cellulose fibres depends on their origin and their morphology.  Mercerisation of secondary wall cellulose ( cotton linters ) Cannot occur if the concentration of NaOH is below 10% (w/w) (Lindgren et al., 1995)  Mercerisation of primary wall cellulose (sugar beet pulp) Starts at concentration of 9% NaOH (w/w) (Dinand <i>et al.</i> , 2002)

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