Abstract

ZAVERI, MITUL DILIP. “Absorbency Characteristics of Kenaf Core Particles” (Under the direction of Dr. Behnam Pourdeyhim). Chopped kenaf core (2” to 4” in length), obtained from Greene Natural Fibers – a company located in Snow Hill, North Carolina, was ground into very fine particles (below 1 mm) and categorized into various size ranges. The ground particles were tested for water absorbency and the optimum particle size, giving maximum absorbency, was determined. Experiments revealed that core of size range 106 – 425 microns gave the highest water absorbency at saturation, up to 12 times its weight. The 425 – 840 micron range was the next highest and it absorbed water up to 10 times its weight. Factors leading to this optimum particle size range were determined, the absorption mechanisms taking place were studied and experimental analysis was done to prove the results obtained. Scanning Electron Microscopy (SEM) images were also taken to understand the shape and profile of the granular particles in fine detail. Various chemical and refining experiments were carried out on the highly absorbent particle sizes (106 – 840 microns) to enhance their bonding properties and to make handsheets from them. The highly absorbent core particles were treated with NaOH in water bath at 90°C, Cooked with NaOH and Na₂S at 170°C in a bomb reactor and treated with water in a water bath at 90°C, all for 3 hours. Handsheets were made from
the chemically treated particles to determine if there was sufficient bonding between them. To enhance the bonding further, the particles were refined in a blender for one hour. The effect of chemical treatment and refining on the absorbency properties of the core was determined. SEM analysis of the particles was done to visualize the fibrillation caused due to refining. Handsheets were made with a blend of hardwood and highly absorbent (untreated and water treated 106 – 840 micron) refined core particles with at least 50% core. The absorption properties of these handsheets were determined and compared with the absorption of a handsheet made from fluff pulp (same basis weight). As the final step, the handsheets made from a blend of core and hardwood pulp were sandwiched between a pair of 17gsm lightly calendared polypropylene spun bond fabrics.
“Absorbency Characteristics of Kenaf Core Particles”

By

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To my parents and my fiancée.
“Absorbency Characteristics of Kenaf Core Particles”

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by

Mitul Zaveri
Biography

Mitul Dilip Zaveri was born on March 19, 1981 in Coimbatore, Tamilnadu INDIA. He received his Bachelors degree in Textile Technology from Kumaraguru College of Technology, Coimbatore, INDIA in April 2002 and also received his Diploma in Textile Technology from PSG Polytechnic, Coimbatore, INDIA in April 1998. He has been a Masters student at the North Carolina State University, in the Department of Textile Apparel Technology and Management, since August 2002.
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Chapter 1 – Introduction

1.1. Introduction to Absorbent Products and their Applications

In the past two decades the disposable absorbent market has grown considerably and has become highly competitive. New products have appeared in the market with newly developed materials and sophisticated structural designs. These products are made from different absorbents or different processes that enhance the absorption capabilities of the material. In spite of all these developments, the field is still in need of an absorbent core that could give good absorbency at a competitive price and have no environmental concerns. A study commissioned by Cotton Incorporated found that 75% of consumers would prefer disposable diapers, and they are willing to pay more for them [1]. Absorbents that are used today are readily available and are used in large quantities in applications like wipes, tissues, paper towels, hygiene products (diapers/sanitary napkins), Oil spills on land and in water, waste water treatment, sewage sludge treatment, separation of oil and water and so on, but all these absorbents have their limitations.

Based on the raw material used to manufacture the product, absorbents are generally classified as Natural absorbents and Synthetic/Polymeric absorbents. An absorbent product usually consists of 2 main components; the first component is the absorbent core (natural and/or synthetic) that is sandwiched between 2 layers of the second component, which
usually is used to give a good feel to the product. In most cases, the second component is made of a textile fiber. This component is made such that it is porous on one side and impermeable on the other. The porous side of the product allows the fluid to get into contact with the absorbent core and the impermeable side prevents the fluid from coming out. The outer layer is also in direct contact with the human body and this makes the feel of the layer a very important issue. The outer layers are made up of soft material that would give adequate feel / comfort to the product. They occupy anywhere between 30 – 50% of the product by weight and the rest 50 – 70% is occupied by the absorbent core.

There are different applications for absorbent products in various fields, absorption of water being the most common, where products like tissue paper, paper towels and diapers are categorized. These materials are made up of natural or synthetic absorbents depending on their end use. Synthetic products are used in hygiene products where very high absorption is necessary and for other applications like wipes, tissue paper etc. natural products like fluff pulp or sphagnum peat moss are used. The usual absorption capability of a fluff pulp ranges from anywhere between 5 – 7 grams for one gram of sample (experiments done). There is always a need for a product that could absorb higher amount of fluid / unit weight, due to the ever increasing cost of pulp.

The other category of application of these products is in the absorption of oil. In any manufacturing firm, oil serves as a lubricant and to clean any spills on land or water, these products are essential. Oil absorbent materials come in different forms. Some of 3 those are granular (powder form) and they also come in the form of socks, pads, towels etc. There has
also been a heavy demand for materials that can separate oil from water and water from oil. The separation of oil from water is done when pure water, free of any contaminants, is required and the reverse is done usually done in petroleum industries where even the slightest of water in the crude oil extracted can cause to major concerns. Absorbent materials that absorb oil from water are completely hydrophobic and the ones that absorb water from oil are completely hydrophilic, so depending on the end use, these products are chosen. In general, these are difficult to find as most of the natural oil and water absorbents found in the market absorb both kinds of fluids which would be considered a failure in this case. In case of oil / water separators it is always better to go for a synthetic absorbent, as they can be completely hydrophobic or hydrophilic, as the need may be.

Wastewater treatment is also becoming a very important issue facing the industry because of increasingly stringent treatment standards being required by the United States Environmental Protection Agency and pending water shortages. Waste water treatment is a field which poses both technical and economic challenges to the industry [2]. Much of the treatment ongoing within the industry is focused toward treating waste water contaminated with organic pollutants, because organic pollutants are common waste constituents. Technologies, such as bio-treatment, air stripping, chemical oxidation and granulated activated carbon have been used with great success [3]. Activated carbon is the most popular choice and it is very effective in doing so, but economically, it is very expensive, especially in applications where the quality of the treated water is not a major concern. Search for an alternate material that gives a relatively good performance in treating waste water and that
can replace activated carbon is always on. Development of a cheaper product would have many practical applications, where the quality of water can be sacrificed to some extent.

1.2. Introduction to Absorbency

Absorbency is a phenomenon characterized by the mode and the extent of transport of liquid into an absorbing material. The main reason for absorption to occur is the driving force that comes from the intrinsic liquid attraction capacity of the material and that is enhanced by a secondary force like gravity or pressure [4].

The term “Absorbency” is used in many different fields with different connotations. For example, in a biological field the term means consumption of a material in a biological or physiological environment. In textiles, absorbency is defined as an interdisciplinary scientific phenomenon that deals with absorption of aqueous fluids either by porous media or by fibrous and polymeric systems. Numerous attempts have been made to define and predict the absorbency using many classical theories, dating back to the 19th century, as well as those developed in the past few decades. But none of them till date has been accepted universally. Many products have been developed over the years that partially fulfill the need of the consumers, but these developments are strictly based on trial and error methods [5].

Absorption is a process where one substance permeates another [6]. The phenomena are generally limited to systems where there is affinity between the liquid and the absorbent. The main driving force for the transport of the bulk of the liquid into a material is the “Capillary Pressure”. Capillary pressure can be defined as the pressure difference existing across the interface separating two immiscible substrates [7].
The absorbing substrate is in most cases defined as a porous medium with interconnected pores of various sizes, where the flow is characterized by the existence of the saturation gradient along the direction of the flow. One of the important aspects of absorbency is the partial dissolution and swelling of the absorbing material. The structure-property relationship of these absorbents is difficult to determine because of the various factors that are to be taken into consideration like hydrophobicity, hydrophilicity, repellency, water resistivity, porosity, swellability etc. This makes it a very complicated issue to predict the amount of fluid an absorbent can absorb and the rate at which the fluid would be absorbed [8, 9].

Regarding the material used for absorbent products, cotton cellulose has been utilized since the time of Egyptian civilization. By the end of 19th century, wood pulp in the form of fluff and then tissue was used as an absorbent material. In the late 1960’s super absorbents came into the picture and they made a great impact on the field. Its absorption capacity was very high due to its amazing water retaining tendency. This helped the industry to come up with more unique, comfortable hygiene products [9].

These super absorbent polymers popularly called SAP’s have the ability to absorb up to 1000 times their weight and that is what made them unique.

The two main problems associated with these super absorbent polymers were

a) They are very expensive when compared to their natural counter-part and

b) The synthetic polymers used in these caused environmental concerns.
1.3. Theories Defining Absorption

Most of the absorption media can be described as porous media. A vast majority of these absorbents contain an inter-connected three-dimensional network of capillary channels of non-uniform shapes and sizes. Fluid flow takes place within extremely complicated microscopic boundaries that make any rigorous solution of the equations of change in the capillary network practically impossible [10].

Absorption can be of two different kinds; a) Due to the pores present within the absorbent. These pores behave like trap zones and hold the liquid in them by forming a chemical bond (usually hydrogen bonds in case of cellulosic materials) causing the material to swell and sometimes by even dissolving cellulose. This is a unique property not found in all the materials and this kind of absorption is termed as Microporous absorption. b) In the second case, the absorption is accompanied by the pores created between the fibers. These fibers might or might not have pores on their surface. The liquid is absorbed in these pores due to the capillaries formed by the random arrangement of fibers and creating a path for the liquid to flow through. This type of absorption is called Capillary absorption and is the most commonly found absorption mechanism. Capillary absorption is applicable to absorbents like fluff pulp, where the fibers are scattered in a random fashion to make a Nonwoven, water is absorbed by the capillaries.

Many of the theories related to absorption are based on the second case, where the liquid is absorbed due to capillaries and more so in the textile field, where they explain how the liquid flows through the pores formed by fibers or yarns when they are brought together.
to make a fabric. In case of woven and knitted fabrics parameters like the direction or the orientation of yarns, the cover in the fabric, yarn count, fiber denier etc., are known. Hence, defining theories to calculate the absorption in a woven or knitted fabric is relatively easy and practical results achieved have shown some correlation with theoretical value, but in the case of Nonwovens, due to the random arrangement of fibers, there is not one particular theory, which explains their absorption capabilities. There have been a few theories that can interpret the absorbency to a certain extent but nothing that can be perfectly reliable.

The absorption mechanism is traditionally interpreted as a flow through a system of capillary tubes using standard capillary flow equations. The unsteady flow of absorbency is studied by using Darcy’s Law. Washburn equation is another theory that gives a good knowledge about the absorbency of a fabric.

1.3.1. Fluid flow through a capillary tube

Absorbent fiber systems with complex pore structures are frequently treated using a simple capillary tube flow model. The liquid moves into a porous medium by capillary pressure i.e., the differential pressure across the liquid-air interface due to the curvature of the meniscus in the narrow confines of the pores. The magnitude of the capillary pressure is commonly given by the Laplace equation as applied to idealized capillary tubes [11].

\[ p = \frac{2\gamma \cos \theta}{r_c} \]

Where, \( r_c = \) Capillary Radius; \( \gamma = \) Surface Tension of the advancing liquid
\( \theta = \) Contact angle at the liquid-solid-air interface
To make it even simpler an idealized tube structure equation, Hagen-Poiseuille law [12, 13] for laminar flow through pipes, can be used. The law states that the volumetric flow rate is proportional to the pressure drop gradient along the tube.

\[
q = \left(\frac{R_c^2}{8\eta}\right) \frac{\Delta P}{L}
\]

\[r_c = \text{Capillary Radius} \quad \eta = \text{Fluid Viscosity}\]
\[\Delta P = \text{Net driving pressure} \quad L = \text{wetted length of the tube}\]

The capillary rise between the time of initial contact and the final equilibrium was obtained by the Washburn equation \([11,12]\).

\[
L = \frac{r_c \gamma \cos \theta}{2\eta} t^{1/2} = k_0 t^{1/2}
\]

\[k_0 = \text{constant}\]

The disadvantages behind these theories is that, in any micro-porous structure, the capillaries are neither cylindrical nor all of them are arranged in parallel. The capillary radius is also not defined as well as the radius is subjected to change depending on whether the capillary are in a dry or wet state. Absorbent textile materials are porous media with structures that are more complex. The pores are interconnected in a three dimensional network and the fluid has to follow through torturous paths rather than a straight line as in capillary tube. In some cases Darcy’s law has been applied in many different fields like, petroleum engineering, soil physics, liquid filtration etc.
1.3.2. Darcy’s law

Darcy’s law is an empirical formula which describes the kinetics of fluid flow through porous media in terms of driving force gradient and the permeability of the medium [13].

\[ q = -\frac{k}{\eta} \frac{\Delta P}{L_0} \]

- **q** = Volume flux in the direction of flow
- **η** = fluid viscosity
- **k** = Permeability of the medium
- **ΔP** = Net pressure head
- **L_0** = Length of the sample in the direction of the flow.

1.4. Properties that Influence Absorption

1.4.1. Pores and porosity

Pores are defined as void spaces distributed extensively throughout the volume of a porous medium. These pores are a three dimensional network through which the liquid flows. The pores in a traditional textile fabric (woven or knitted) are usually two dimensional because they have a very negligible thickness, but in the case of a Nonwoven fabric, especially high-lofts, this property is a pure three dimensional property where the thickness plays a major role.

Porosity can be defined as the ratio of the void space in a porous medium over the total bulk volume of the medium. It is a dimensionless quantity between 0 and 1. If the porous medium is made up of regularly packed pores of uniform size and shape, the porosity can be calculated from purely geometrical consideration like in the case of woven fabrics.
Assuming there is no change in dimension, if the entire pore space is filled up with the absorbed liquid, the porous sample is said to be fully saturated and the maximum absorption capacity of the sample can be defined as the mass of liquid absorbed per unit mass of dry solid medium.

There are 3 factors related to pores that would affect the absorption capacity of the material. The pore volume affects the total volume of fluid absorbed and thus the ultimate absorbency of the fabric, the pore size and pore distribution affects the rate at which the fluid is absorbed. The pores can be generally classified into internal and external pores. The external pores are the ones that form the capillaries and the internal pores are the micro-pores available in the material. Usually the internal pores absorb relatively lower amount of fluid when compared to the external pores or the capillaries. Porosity is the most important property that affects the absorption capacity of the material.

1.4.2. Chemical structure of the absorbent

Any cellulosic material has a tendency to absorb fluid due to its chemical structure and the hydroxyl groups available in their chain. The hydrogen and oxygen bonds are at the outer surface and immediately absorb any amount of water present forming a hydrogen bond. Most of the natural absorbents present today are made of cellulosic fibers like rayon, cotton, fluff pulp and Kenaf Core.

The chemical structure has direct effect on the absorbency characteristics of a product. Liquid cannot penetrate into a crystalline region as easily as it can penetrate into an amorphous region, i.e. an amorphous region would initiate liquid absorption better than a
crystalline region. The rate of absorption to an extent also depends on the how amorphous or crystalline the material is. The chemical structure also explains how the absorbent will be attracted towards different liquids. The chemical structure determines the hydrophobicity or hydrophilicity of the material. Another property that is influenced by the chemical structure is its swelling. Amorphous parts of a cellulosic material, depending on the interactive liquid being used, gets dissolved on contact and this causes the liquid to penetrate further inside the material causing more swelling and this is the reason of expansion that occurs during absorption. If the interactive fluid is water, only the parts of the amorphous regions will be dissolved and will allow hydrogen bonds to be created, whereas the parts of the crystalline region will not allow water to get into them.

A simple chemical structure of cellulose is shown below and it can be seen how the hydroxyl groups are available to absorb water.

![Fig.1](image-url)
1.4.3. Viscosity of the liquid

The viscosity of the liquid to be absorbed determines the rate of fluid absorption to a greater extent than the ultimate absorption capacity of the material. A fluid with a higher viscosity takes a higher amount of time in penetrating through a pore when compared to liquids that are less viscous. Another problem faced with viscous liquids is that, it is very difficult for the fluid to penetrate into fine pores in the absorbent. This was not considered a factor in the research conducted because the only liquid used was water.

1.4.4. Surface tension and wettability of the liquid

Surface tension of a liquid has an affect on the wettability of the absorbent. Wetting is a process by which a fiber-air interface is replaced by a fiber-liquid interface and the amount of liquid transferred into the absorbent is measured by the measuring the advancing contact angle [14]. A liquid with higher surface tension has a low uptake rate. To increase the rate of uptake, the surface tension of the liquid is lowered by adding surfactants. The lowering of surface tension may also cause decrease in the rate of uptake, if the advancing contact angle is not decreased. This advancing contact angle is directly related to the wettability and an increase in the advancing contact angle means an increase in wettability [12].

1.4.5. Surface area of the absorbent

Surface area affects the absorption of the material to a certain extent and more so in case of non-porous media. Usually as the surface area increases, it increases the capillaries that are being formed and hence increases the absorption, but as determined in this research,
surface area has a smaller impact on absorption properties of material that are not completely solid and have micro-pores in them.

1.5. Types of Absorbents and their Applications

In terms of water absorbency there are not many alternatives being used as the absorbent core. Presently wood pulp is the most popular absorbent core and it is used in the form of fluff pulp in products like wipes, tissue papers and paper towels. This is because of its cost, availability, good overall absorbency (6 to 7 times its weight) at a decent rate. It is also being used in diapers in combination with synthetic absorbents. The most important advantage of wood pulp over synthetic polymers like Super Absorbent Polymers is, they are natural, biodegradable and they do not have environmental issues. Synthetic polymers, that absorb fluids, are not environmentally friendly and there is always a search for a better absorbent than wood (fluff) pulp. The ever rising cost of fluff pulp is causing concerns and increasing the need for a better and cheaper, natural, biodegradable absorbent core. There are an innumerable number of polymeric/synthetic products in the market that are used as absorbents to clean oil spills and for other environmental conservation purposes like wastewater treatment. The products usually available are melt-blown polypropylenes, Super Absorbent Polymers (SAP’s) etc. The SAP’s are said to absorb 1000 times their weight, where as wood pulp absorbs only about 8 to 9 times its weight. But the prices of the SAP’s are too high for its use in day to day applications.
Chapter 2 – Research Objective

a) To find the optimum particle size of Kenaf Core that gives the maximum absorbency.
b) To make a handsheet from the optimum particle size.
c) To find ways to enhance the bonding to make the handsheet.
d) To sandwich these handsheets made from highly absorbent Core particles between a pair of spun-bond fabrics.
e) To understand the capillary and micro-porous absorption mechanisms.
Chapter 3 – Literature Review

3.1. Introduction to Kenaf

Kenaf is a 4,000 year old crop with roots in ancient Africa. A member of the hibiscus family (Hibiscus cannabinus L.), it is related to the family of cotton and okra, and grows well in many parts of the United States. Kenaf grows quickly, rising to heights of 3.5 – 4.5 meters (12 to 14 feet) in as little as 4 to 5 months. The U.S. Department of Agriculture studies show that Kenaf annually yields of 6 to 10 tons of dry fiber / acre, which is generally 3 to 5 times greater than the yield for Southern pine trees. Kenaf is generally planted in May and harvested in March after drying in the field during the winter months. At the end of the growing season, the kenaf plant flowers. After blooming the flower drops off, leaving a seed pod behind [15].

Kenaf grows well in most parts of the United States and requires few or no pesticides depending on where it is grown. Due to its fibrous stalk insects rarely cause damage. Few chemicals are needed to grow kenaf, but to ensure good soil conditions, some fertilizer and a single herbicide treatment may be used to control weeds. Although kenaf is adaptable to various soils, it grows best in well-drained, sandy loam soil [16].

Kenaf has a relatively wide range of adaptation to climate and soils. With the exception of some early types developed for the Asiatic regions of the former USSR, most of
the current kenaf varieties and technologies favor growing at low elevations between 37° N and S latitudes. Optimum growth is generally found in areas like the lower Rio Grande Valley of South Texas with its long, warm growing season and moderate rainfall backed up by irrigation [16]. In the 1970’s after examining over 500 species of annual plants, it was concluded that Kenaf was the most viable alternative fiber source for the pulp and paper industry [17]. Kenaf bast fiber takes 15 – 25 % less energy to pulp than the commonly used Southern White Pine. Kenaf paper is stronger, whiter, durable and more resistant to yellowing and has better ink adherence to tree paper [18].

The kenaf plant is made up of 2 components. The outer periphery is made up of long fibers called the Bast fibers and it occupies around 30 – 40% of the plant by weight. The inner woody component is known as the Core and it weighs around 60 – 70% of the plant. The harvested kenaf plants comprises of the whole stalk (Core and Bast) together. They are bound together by lignin. To separate these 2 components a process called retting is done. There are basically 3 types of retting,

a) Bacterial Retting
b) Chemical Retting
c) Mechanical Retting

In Bacterial retting the whole stalk kenaf plant is immersed in water at room temperature for a period of 5 to 22 days. During this period, due to bacterial action in the whole stalk, the lignin gets dissolved and the bast fibers are separated from the core.

Chemical retting is a process done to achieve the separation of bast and core rapidly. In this process the whole stalk kenaf is placed in an alkali bath for a period of one or two hours. The alkali dissolves all the lignin and separates out the fibers from the core. The fibers
are washed after the process and are immediately neutralized. The chemical retting changes certain properties in the fiber and the core due to the chemical action on the fibers. There is a loss in tenacity, lusture and color. It also gives low strength and elongation when compared to the fibers that are obtained by bacterial retting.

Mechanical retting is a process to separate the core and the bast fibers by mechanical action. This type of retting is very cheap and gives a very high yield as there are no chemicals involved and there is no loss in strength as well. The disadvantage of this process is that the bast fibers remain stiff and brittle and are very difficult to process especially in textile machinery where the fibers are required to be flexible. The stalks are left in the field to allow bacterial or fungal action to take place that loosens the bast from the stalk. This stalk with loose bast fibers on it is subjected to beating process that separates the fibers into two separate parts, the bast and the core. Mechanical retting is practiced in places where bacterial retting cannot be done due to the lack of resources like a pond or a water bath to soak the stalks in water for a period of 20 days. Once the bast fibers are separated from the core they are used for different applications as they have different properties.
[Table 1] Chemical Composition of Kenaf Bast fiber

<table>
<thead>
<tr>
<th>Constituents (%)</th>
<th>Natural</th>
<th>BR</th>
<th>CR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cellulose</td>
<td>59.8</td>
<td>73</td>
<td>82.8</td>
</tr>
<tr>
<td>Hemi Cellulose</td>
<td>11.6</td>
<td>12.6</td>
<td>8.2</td>
</tr>
<tr>
<td>Lignin</td>
<td>17</td>
<td>5</td>
<td>3.6</td>
</tr>
<tr>
<td>Cell Wall Contents</td>
<td>10.4</td>
<td>7.9</td>
<td>4.6</td>
</tr>
<tr>
<td>Ether Soluble Extract</td>
<td>1.2</td>
<td>1.5</td>
<td>0.8</td>
</tr>
</tbody>
</table>

BR – Bacterial Retting  CR – Chemical Retting

[Table 2] Chemical Composition of Kenaf Core fiber

<table>
<thead>
<tr>
<th>Constituents (%)</th>
<th>Natural</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cellulose</td>
<td>37.6 – 49.0</td>
</tr>
<tr>
<td>Lignin</td>
<td>15.0 – 21.0</td>
</tr>
<tr>
<td>Sugars</td>
<td>18.0 – 24.0</td>
</tr>
<tr>
<td>Ash</td>
<td>2.0 – 4.0</td>
</tr>
</tbody>
</table>

The bast fiber is like a conventional textile fiber obtained in bundles and can be cut into a specific length. These fibers are coarse and brittle in nature and are made soft and flexible by adding alkali in water emulsions. The density of the bast fiber is $1.293 \pm 0.006$ gm/cm³ [19]. They were traditionally used in making Cordage yarns, Canvas, Sacking, Carpet backing, padding lignocellulosic composites, door and instrument panels. More recently they are being used as a noise insulation material and fiber mat in automobiles. Researchers at Kansas State University have been trying to make fabrics from a blend of Kenaf fiber and Cotton. In spite of all these uses, the maximum research done on Kenaf bast is on making high quality paper. The Kenaf bast offers a very good alternative for making specialty paper and has excellent properties in terms of papermaking.
The Core on the other hand is a hard wood like material and is mainly used in absorbent applications like potting mixes, packing material, as an organic filler, additive for drilling muds, for insulative and Acoustic Pads, erosion mats, animal bedding and some wood based composites. The density of the core is 0.09 – 0.11 gm/cm³ and the length of refined fibers is 0.6 mm [20]. Heavy research is going on in making Kenaf Core a natural, bio-degradable oil absorbent. Kenaf Core is said to absorb 5 times its weight of oil. A few companies in the United States like Kengro Corporation, S & S farms and Greene Natural fibers are conducting research on Kenaf Core for these applications.

There is another component present at the innermost part of the kenaf stalk. This component is called the Pith. There has not been any literature reported on the pith, but it is said to be a highly absorbent material that can absorb up to 20 times its weight but not much literature is published on it so far. Pith is very light weight and compressible with a density lower than the bast and the Core. The other advantages of kenaf are it is biodegradable and thus environmentally safe. It is rapidly renewable. It requires few or no pesticides for its growth.

3.2. Related Work

This research work focuses on the water absorbency characteristics of Kenaf Core particles of size less than 1000 microns. Core occupies 60 – 70% of the Kenaf plant and it increases in the plant with time. The plant is basically grown for its bast fibers that are sold to the paper makers in general or more recently to some Nonwoven and Composite industries. Core being the major component goes for absorbent applications such as animal bedding,
potting mixes, wood based composites and so on. There is lot more potential in the Core because of its micro-porous structure. In addition to the micro-pores in its structure, the material comprises, approximately 50% by weight, of cellulose which renders the material highly absorbent. There has been a lot of research done in terms of evaluating the absorbency of the Kenaf Core with respect to different kinds of oils.

3.2.1 Study on Kenaf Core by Naval Facilities [20]

The Naval Facilities Engineering Service Center (NFESC) has evaluated kenaf core as an absorbent for cleaning oil spills. This research was done in March 1999. The Core obtained was reduced in size by using hammer mill and then filtered using screens into two particle sizes ranges, between 1/4” to 1/8” (3.175 – 6.350 mm) and less than 1/8” (less than 3.175 mm). The particles were compared against the commercially available absorbents, peat moss, kitty litter and with standard polypropylenes. T-201 Heavy crude, #2 Diesel and T-102 Light Crude were the different kinds of oils used for this experiment. The results indicated that the Core particles outperformed the two other natural absorbents by a significant margin but was below when compared to the polypropylenes. The study also suggested that Kenaf fines (particles smaller than 2mm) would give absorbency comparable to the polypropylenes. The table below shows the comparison between the absorbency of Core with the leading natural absorbents (Peat moss and Kitty litter) and polypropylenes.
[Table 3] Absorbency Ratios for Kenaf and other Sorbents

<table>
<thead>
<tr>
<th>Oil Type</th>
<th>Kenaf Milled # 1</th>
<th>Kenaf Milled # 2</th>
<th>Peat Moss</th>
<th>Kitty Litter</th>
<th>Polypropylene¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-201 Heavy Crude</td>
<td>2.9</td>
<td>1.64</td>
<td>1.23</td>
<td>0.31</td>
<td>9.81 – 15.21</td>
</tr>
<tr>
<td># 2 Diesel</td>
<td>4.39</td>
<td>5.54</td>
<td>3.5</td>
<td>0.44</td>
<td>9.22 – 13.16</td>
</tr>
<tr>
<td>T-102 Light Crude</td>
<td>5.87</td>
<td>6.92</td>
<td>3.48</td>
<td>0.45</td>
<td>9.81 – 16.61</td>
</tr>
</tbody>
</table>

¹ – Ranges based on results of the lowest and highest performing Polypropylenes.
Kenaf Milled # 1 = 1/4” to 1/8” Kenaf Milled # 2 = less than 1/8”

3.2.2. Natural Sorbents in oil spill clean-up [21]

Chol et al. conducted a research on different natural sorbents that absorb oil and that can be used in an oil spill cleanup applications. The various absorbents taken into consideration were Milkweed fiber, Cotton fiber, Polypropylene fiber and web and Kenaf Core. The results stated that the oil absorption capacity of Kenaf Core was comparable to the polypropylene web, when the liquid to be absorbed was high viscosity bunker C oil. The procedure was mainly designed to evaluate all the above contenders for an oil spill in water. A sample of artificial sea water was mixed with a predetermined amount of oil in a beaker and 1 gm of sample was placed in the system and the contents in the vessel where shaken for 10 mins at a constant rate. The wetted absorbent was weighed after being drained. The amount of oil sorbed by the sorbent was determined by subtracting the water content and the initial sorbent weight from the total weight of the wetted sorbent. The quantity was recorded in terms of grams of oil absorbed per each gram of sorbent. Recovery of sorbed oil and reusability of sorbents was also studied.
This experiment also proves that Kenaf Core can be used in applications where separation of oil from water is necessary as Kenaf Core shows a tendency to absorb oil when it is present in a water-oil mixture.

3.2.3. Research conducted by S & S Farms [22]

S & S farms is a company that manufacturers 100% natural and bio-degradable oil absorbent products. They conducted a research that compared Kenaf Core, Peat moss and Clay on T-102 light crude oil. In the experiment conducted, a fixed amount (42 gallons) of petroleum based product (T-102 light crude) was placed in 3 separate barrels. The sorbents were added in measured quantities into the barrel until all the oil was absorbed. Results indicated that only 25 pounds of kenaf was necessary to absorb all the T-102 light crude oil, when compared to 67 pounds of peat moss based product and 665 pounds of clay based product.

![Fig. 5](image)
The research also indicated that there were several other hydrocarbons absorbed by the product made from Kenaf Core like, Acetone, vegetable oils, benzene, kerosene and other hydrocarbons that can contaminate water. It was suggested that future work can be done on studying the properties of Kenaf Core in waste water treatment.

### 3.2.4. Research by Greene Natural Fibers on oil absorbency [23]

Greene Natural is a company located in Snow Hill North Carolina that grows Kenaf and has facilities to separate the Bast and Core fiber. They have conducted a research experiment on Oil absorbency of Kenaf and compared it to commercial available absorbents like, Sphagnum peat moss, Bentonite, Gypsum, high absorbent polypropylene and cellulose fiber. Results revealed that Core was the second best, next to highly absorbent polypropylene webs in terms of oil absorption. The level of oil absorbency of Kenaf Core was more than twice when compared to the other natural and cellulosic products and almost as good as highly absorbent polypropylene.

![Oil Absorbent Material Performance](Fig. 6)
3.2.5. Research by Greene Natural Fibers on moisture absorbency [24]

Greene Natural Fibers also conducted a research on Moisture absorbency of Kenaf Core. The other contenders that were taken into consideration for comparison were wood fibers, pine and cedar. Results revealed that Kenaf Core had the highest moisture absorption in comparison with wood, pine and cedar.

![Bedding Material Absorption](image)

**Fig. 7**

3.2.6. Evaluation of Kenaf Core as a desiccant [25]

The department of Animal and Dairy science at Mississippi State compared Kenaf core and silica gel to determine its suitability as a packaging desiccant. Three sample types were achieved by using kenaf core flour, kenaf core bin 3, and silica gel beads. Results suggested that silica gel absorbed more moisture than core, but core may be a suitable organic alternative to silica gel. At 94% R.H core absorbed moisture up to 27 – 28% of its weight. Core absorbed higher amount of moisture at high humidity levels. It was also
suggested in the study that the low density of core could provide moisture absorbency at a lower cost.
Chapter 4 – Commercially Available Water Absorbents

4.1. Fluff Pulp

Fluff pulp is manufactured from wood and is the most widely used natural absorbent core in wipes, hygiene applications and all other absorbent products. It is a light, bleached, absorbent mixture of chemical and/or mechanical pulp and is used as a filler in sanitary products such as disposable diapers, bed padding, napkins, bandages, and other personal hygiene products. Fluff pulps are generally made from softwood fibers to give it a soft feel. Soft wood fibers are long and are well suited for high fluid transport, unsurpassed absorbent capacity and exceptional strength in case of air laid structures. Fluff pulp is superior in absorbency, as it is basically a cellulosic material (wood based) and the pulping action enhances their tendency to form a large amount of capillaries giving a higher absorption. In some cases, debonders are added to the pulps to reduce their strength, giving a sheet that is softer and easier to defiberize, resulting in increased absorption due maximizing the amount of free fibers and minimizing the formation of fiber bundles [26].
4.2. Sphagnum Peat Moss

Sphagnum peat moss is another 100% natural organic matter made up of partially decomposed aquatic moss that accumulates in the lower levels of a peat bog. Its large cell structure enables it to absorb air and water like a sponge. Sphagnum peat moss is well suited for indoor and outdoor horticultural applications. It is used as a soil amendment for gardens, for starting seeds, potting hanging baskets, planting perennial beds, growing vegetables, and planting or transplanting shrubs and trees. It’s also great for preparation and restoration of lawns and for composting. Peat products retain up to 8-10 times their weight in moisture. Peat forms at a rate of 1 to 2 millimeters per year [27].
Chapter 5 – Materials and Methods

The raw material (Kenaf Core) was obtained from Greene Natural Fibers, a company located in Snow Hill, North Carolina. Core in its as received form was chopped in pieces of up to 4” in length. From the literature review it was decided to grind them into various particle size ranges (below 1000 microns) and test them for water absorption. The kenaf plant was retted in the fields, followed by mechanical separation of the bast and the core through repeated beating action. The final core material obtained was 90% pure containing some pith and bast fibers. The pith component is a sponge like material located at the central part of the stalk. It is said to be highly absorbent but there is not much literature is reported.

5.1. Evaluation of Optimum Core Particle Size

The chopped core was ground in the willey mill and ball mill, available in the Pulp and Paper Science Department at the NC State University. The ground particles obtained from the mills were categorized into various size ranges by shaking them in an agitator for five mins and then manually sieved until there were a negligible amount of particles falling off the sieve.
5.1.1. Willey mill

Willey mill is a machine that is used for grinding dry wood pieces or chips that are used in papermaking. It is available in the Pulp and Paper Science Laboratory in the NC State University.

It consists of a heavily built rotating blade that revolves at high speed. The outer periphery of the blade and the inner casing of the machine have very little space in between them that causes the grinding action. Depending on the size of the sample fed and the amount of grinding action required, the space between the edge of the rotating blade and the inner casing is adjusted. The chopped Core was fed in batches of approximately 50–75 grams. A metal plate was placed before the contact of the material with the blades, to allow gradual feeding. As the material comes in contact with the blades rotating at high speed and due to the lack of space it is broken down into small pieces. This action keeps repeating until the particles are small enough to fall through the screen placed below the blades. There are 4 different sizes of screens, placed at the bottom of the mill. The screens hold the particles and keep them in contact with the rotating blade until they are small enough to fall through. The smallest screen gives an approximate size of around 100 microns and the largest screen gives an approximate size of about 1000 microns. Different particle size ranges were collected using different screens.

The particles were then categorized into the required particle size range using sieves obtained from Fisher. The sieves along with the ground material were placed on an agitator
which shook the sieves randomly for five minutes. The particles were then manually sieved until the particles falling from the sieve were negligible.

5.1.2. Ball mill

To obtain particles below 100 microns a ball mill was used. A ball mill consists of a motor that turns two drive shafts on which the container with the sample is placed. The container contains Lead-Antimony balls that grind the particles into a fine dust. Ball mills are generally used to grind glass, making ceramic glaze and powdering various other substances. They can also be used to grind any coarse particles into fine powder. The mill used for this experiment was also obtained from the Department of Pulp and Paper Science, NC State University.

The 3 factors that affect the amount of grinding done on a Ball mill are a) The number of lead-Antimony balls used, b) The amount of running time of the mill and c) The size of the input particles. In this case, instead of lead-antimony balls, glass balls were used and the particles fed were in the order of less than 1000 microns. During the first experiment the mill was run for a period of 24 hours with 100 grams of sample in the container. The input sample size was between 250–840 microns. The powdered core obtained was categorized into two size ranges, below 38 microns and 38-106 microns. There were negligible quantities of particles above 106 microns after the grinding treatment and they were removed. The second experiment conducted was to obtain particles between the ranges of 106 – 250 microns as it was very difficult to obtain these from the willey mill. 100 grams of particles between 250 and 840 microns were again placed in the same container with the
glass balls and the running time was reduced from 24 hours to 6 hours. It was found that even after reducing the time to 6 hours the particles obtained were below 106 microns. Hence a third experiment was carried out and the particle size that was fed was raised to the range of 425 to 840 microns and the ball mill was set to work for only one hour. The particles obtained were approximately between 106 and 425 microns and a very few fines of below 106 microns were obtained.

The various particle size ranges obtained were as follows,

- Θ Below 38 microns
- Θ 38 – 150 microns
- Θ 150 – 250 microns
- Θ 250 – 425 microns
- Θ 425 – 840 microns
- Θ Above 840 microns

Once the size ranges were obtained they were tested in the NCRC testing device for absorbency.

**5.1.3. NCRC Absorption Testing Device**

Absorbency tests were carried out on the NCRC testing device that resembled the Gravimetric Absorbency Testing System (GATS), available in the NCRC physical testing lab. The device was designed to measure absorbency from an unlimited liquid reservoir. The instrument works on the principle of gravity and a demand wettability concept, allowing a sample to draw as much liquid as it can hold. The testing instrument gives an electrical readout of weight (in grams) of liquid absorbed into the sample. A plotting mechanism is attached to the computer that plots the absorbency as a function of time. The frequency of the time interval can be varied in steps of up to 1/10th of a second. The fluid reservoir is filled
with the liquid to be absorbed, until it reaches a minimum point. A 3mm diameter tube transfers the fluid from the reservoir to the sample holder that accommodates a porous plate in the slot provided. The porous plate is approximately 3¼” in diameter and has a depth of about 1”. Porous plates with varying pore sizes are available to allow the device to be used for different liquids.

Once the required amount of fluid is filled in the reservoir, the height of the sample holder is adjusted to be in line with the level of fluid in the reservoir to achieve the “zero hydrostatic head pressure” that was desired. Depending on the kind of test to be performed, a positive or a negative hydrostatic head can be used. As the top of the porous plate is adjusted to be in level with the fluid in the reservoir, due to the Zero hydrostatic head, the liquid flows from the reservoir to the porous plate and spreads itself on the surface of the porous plate. The required sample to be tested for absorption is weighed and the weight is recorded in the NCRC testing device. The device is then setup for the required absorption time and the timing interval. It is interfaced with a recorder which records the loss of water in the reservoir and provides a plot of “Absorbency vs. Time”. For every gram of water absorbed, the level of the sample holder is brought down by a fraction to keep in line with the level of water in the reservoir which constantly decreases due to the water uptake by the particles. The reservoir is re-filled by a solenoid valve that can be operated when necessary.

The first sets of experiments were conducted with water and two gram samples for duration of 1800 seconds. After 500 seconds the samples attained saturation and the increase in absorption was negligible, so the absorption time for all future tests was reduced to 600
seconds. Due to the small size of these particles, the porous plate was thoroughly cleaned after every test to avoid clogging of the porous plate. A minimum of 5 tests were done for every sample and the mean and standard deviation was recorded.
Willey Mill

Feeding Zone

Metal flap (ensure gradual feeding)

Fig. 8

Rotating Blades

A close view of the blades and sieve

Inner casing of the Mill

Fig. 9

Rotating Blades

Sieve

Ground particles - Output
Ball Mill

Fig. 10

Ball mill with sample and lead-antimony balls

Shaft that rotates the mill
Gravimetric Absorbency Testing System (GATS)

Full scaled view – GATS

Fig. 11

Top view – Sample Holder

Fig. 12
5.2. Producing Handsheets

One of the primary objectives of this research was to make handsheets from the highly absorbent core particle sizes and sandwich them in between spun-bond fabrics. Handsheets with particles below 106 microns could not be made as the mesh on the available handsheet maker was of 106 microns and anything smaller than that would go through the mesh. Handsheets were made only from the highly absorbent particles (106 – 840 microns).

A handsheet can be defined as a sheet of fibers that are oriented in a perfectly random fashion. These handsheets are produced by an instrument called the handsheet maker, which is generally used to make paper samples. This instrument was available in the Pulp and Paper Science Department, NCSU.

5.2.1. Moisture Analyzer

Moisture analyzer is a testing instrument used to analyze the amount of water retained by the sample at any given time. This instrument was extensively used in this research as the samples were tested for their moisture content before handsheets made from them. This was done because the basis weight of a handsheet is calculated on an Oven Dry (OD) basis. The instrument was also used to calculate the proportions of water held in the micro-pores of the core particles after they were centrifuged.

Moisture Analyzer is a testing instrument that weighs the input sample at standard temperature and relative humidity. The sample is placed on a weigh pan and the initial weight is recorded. The lid over this weigh pan is closed to avoid any interference of air. The
instrument heats the sample up to a temperature of around 160ºC. This process removes all the water and moisture present in the sample. The loss of weight from the sample is recorded as the final weight. The difference between the initial and the final weight gives the amount of water that was present in the micro-pores of the input sample. The output is expressed as a percentage of the input.

\[
\% \text{ of moisture within the material} = \frac{\text{Weight of (input sample} - \text{output sample)}}{\text{Weight of input sample}} \times 100
\]

### 5.2.2. Handsheet-maker

Handsheets are a sampling instrument used in the Pulp and Paper industry to make paper samples from softwood / hardwood pulp. The uniqueness of this machine lies in preparing samples (handsheets) that are completely random and of negligible thickness. The conventional handsheets are usually made of a minimum of 1.2 OD grams of pulp and the diameter of the handsheet is roughly 6 inches. The handsheet is made by dispersing a predetermined amount of fibers in water. To begin with, the required amount of un-dried pulp is passed through a pulp disintegrator. The pulp disintegrator is an instrument that breaks down the pulp (originally in the form of small wet pieces into long fine fibers) and forms a perfectly random distribution of fibers in water. Depending on the sample, the pulp disintegrator is set for a predetermined number of rotations. Once the solution is obtained, it is added to the handsheet maker and the rest of the handsheet maker is filled with water. The fibers are mixed well with the help of a perforated stirrer. A valve is released that drains out all the water, due to gravity, leaving the fibers on the mesh of the handsheet maker. Due to
the random distribution of fibers in the solution, a perfectly random sheet without any orientation in one particular direction is obtained. The wire mesh can be of different sizes and in this case, a 106 micron mesh is used. The fibers on the mesh are bonded together due to hydrogen bonding between the fibers.

This sampling technique might serve as a disadvantage in some cases because of the random fiber orientation, as in the real case (industry) the fibers tend to orient themselves in the machine direction and the paper or fabric obtained from a full scale machine is not as isotropic as a handsheet.

5.2.3. Procedure

1) Take a minimum weight of 1.2 Oven Dry (OD) grams of fibers.
2) Agitate the material in the Pulp Disintegrator for five minutes (depending on the sample) to avoid agglomeration of fibers.
3) Add the solution from the Pulp disintegrator into the handsheet maker and fill the rest of the handsheet maker with water and mix the contents with a perforated stirrer.
4) Release the lever to remove all the water from the handsheet maker, due to gravity and collect the fibers on the mesh.
5) Place a blotting paper on the handsheet and apply pressure by rolling a heavy metal piece on the blotting paper to attach the handsheet to the blotting paper.
6) Lift the blotting paper along with the handsheet.
7) Dry the handsheet in a drier and weigh immediately to calculate the percentage weight loss (if any).
Handsheets were made from untreated core particles, but due to insufficient bonding between the particles, they simply scattered themselves on the mesh of the handsheet maker, like sand, and did not form a handsheet. As a handsheet from 100% core was not possible, handsheets were made from 50/50 blend of hardwood pulp and core. A handsheet was produced that had two distinct regions due to the insufficient bonding between the particles. The results of the handsheets obtained from various particle sizes are explained in detail in the results and discussion section.
Fig. 15

Fig. 16

Fig. 17
5.3. Enhance Bonding

As 100% untreated core handsheet was not possible the handsheet made from a 50/50 blend of core and hardwood pulp gave a rough handsheet with 2 distinct regions, the core were subjected to some chemical and mechanical treatments that would assist in enhancing their bonding properties. Only the highly absorbent core particles (106-250, 250-425 and 425-840 µ) were subjected to these treatments.

5.3.1. Chemical treatment

Kenaf bast fibers have been very strongly recommended for making paper and they are said to be the best replacement for wood fibers to make high quality paper. There is also abundant literature that describes various methods to manufacture paper from core. Paper manufacturing requires fibers to bond well with each other which gives strength to the final product. To achieve the required bonding the wood chips are pulped. During the pulping process, the wood fibers are treated with Sodium Hydroxide (NaOH) and Sodium Sulphide (Na$_2$S) at high temperature (90ºC – 170ºC).

The pulping process causes,

a) Increase in hydrogen bonding between the fibers.

b) Swelling of fibers and

c) Removal of lignin (which is hydrophobic, thus increasing their water absorption capability).
The objective of this experiment was to obtain a 100% handsheet from core with relatively less or no loss in absorption. The various details required to prepare these experiments were available in the pulp and paper science literature.

Three sets of experiments were conducted using three solvents (NaOH, Na₂S and water) and two heating instruments (water bath and bomb reactor). A constant time of three hours was fixed for all experiments and the temperature was varied from 90°C in water bath up to 170°C in a bomb reactor.

[Table 4] Chemical Experiments

<table>
<thead>
<tr>
<th>Ex No.</th>
<th>Chemicals</th>
<th>Heating Method</th>
<th>Temp (°C)</th>
<th>Liquor Ratio</th>
<th>Sample Size (OD grams)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cooked</td>
<td>NaOH (15%) and Na₂S (10%)</td>
<td>Bomb Reactor</td>
<td>170</td>
<td>15:01</td>
<td>30</td>
</tr>
<tr>
<td>Alkali</td>
<td>NaOH (15%)</td>
<td>Water Bath</td>
<td>90</td>
<td>15:01</td>
<td>30</td>
</tr>
<tr>
<td>Water</td>
<td>None (Soaked in Water)</td>
<td>Water Bath</td>
<td>90</td>
<td>15:01</td>
<td>30</td>
</tr>
</tbody>
</table>

Particle sizes: 106-250, 250-425 and 425-840 microns
Time: 3 hours

5.3.1.1. Water bath

Water bath is a simple instrument used for heating samples to a temperature as high as 90°C (experiments 2 and 3 in this research). The sample to be heated is sealed in a plastic cover with the chemicals. The temperature of the bath is raised to 90°C in 30 minutes. The sealed plastic cover with the sample is placed in the water bath for three hours. After heating the samples were placed on a cheese cloth and squeezed to drain out the excess water.
5.3.1.2. Bomb reactor

Bomb reactor is a large machine that is used to pulp wood chips to make paper. The machine comprises of 6 bombs made of iron. These bombs are placed in the slots of a rotating reactor that rotates at a very slow speed to circulate the chemicals around the samples. A steam valve is provided to apply pressure in the bomb. If the sample is to be cooked under no pressure then the valve is left open and vice-versa. The initial temperature of the bombs is 20°C and is raised to 170°C within a period of 25 minutes. The highly absorbent core particles (106 – 250, 250 – 425 and 425 – 840 microns) were subjected to the cooking treatment in the bomb reactor. Once the experiment was completed, the bombs were removed from their slots and placed in cold water for a period of 15 minutes before extracting the cooked samples.

5.3.1.3. After treatment

After the chemical treatment, the particles were extracted from their respective machinery and washed in a Pulp washing equipment. The samples are placed on the equipment and washed manually by pouring water on the particles. The chemicals and water is sucked out through vacuum. The samples are washed thoroughly, centrifuged and air dried before further experiments.

The treated particles were tested for enhancement in bonding by making handsheets from them and testing was also done to determine if there was any difference in the absorption levels of treated particles. The results are reported in the results and discussions section.
Fig. 18

Water Bath

Samples placed inside the bath

Temperature Control

Fig. 19

Pulp Washing Equipment
Bomb Reactor

Full scale Image – Bomb Reactor

Fig. 20

Steel bombs placed in their slots

Steel Bombs

Fig. 21

Fig. 22
5.3.2. Refining

After the highly absorbent core particles were chemically treated, handsheets were made from them to see if there was sufficient bonding. A 100% core handsheet was still not possible and a 50/50 blend of hardwood pulp and treated core produced a handsheet that showed the same results as the ones obtained with untreated particles (a rough handsheet with two distinct layers). The detailed absorption and bonding results are reported in the results and discussions section.

To further enhance the bonding between the core particles a process called refining was done. Refining is done to open the pulped fibers by mechanical fibrillation, hence creating more binding points. It enhances hydrogen bonding by increasing the number of contact points between the fibers. Refining can cause reduction in length or width of the particles due to the shearing action.

There are many ways a fiber can be refined, the ones that are generally used are single or a double disc refiner or a blender. As the particles to be refined were too small (between 106 – 840 microns) when compared to the fibers generally used to make paper (2-4mm), blender was used as the refining instrument.

5.3.2.1 Blender

The blender is a cylindrical vessel and has a sharp blade at its base and rotates at high speed. The rotating action of the blade generates a shearing action that causes the particles to fibrillate. The core particles were refined in the blender at 20% consistency (20 OD grams of core in 1000 grams of water) and refined for an hour. The refined core particles were washed
in the pulp washing equipment. The particles were then centrifuged and air-dried before further experiments.

The refined core particles were tested for absorbency on NCRC testing device and handsheets were made to test if there was any enhancement in the bonding between particles. Cooked and alkali treated particles, in the range of 106 – 840 microns, showed a loss in their absorption property and hence were not considered for the refining experiments. 6 samples of untreated and water treated highly absorbent particles (106-252, 250-425 and 425-840) were refined. The results of absorption and bonding tests of refined particles are reported in the results and discussion section.
Fig. 23

Fig. 24

Blade inside the blender
Chapter 6 – Results and Discussions

The first absorption testing was done with the untreated core samples ranging from below 38 microns to above 840 microns. All samples were tested for water absorbency in the NCRC absorption testing device. All absorption tests were done for a minimum of 5 runs and the mean and standard error were plotted.

6.1. Absorption of untreated Core Particles

The above graph shows, after a period of 1800 seconds in the GATS, there are two size ranges (106 – 250 and 250 – 425 microns) that show maximum absorbency.
(approximately 12 grams of water / one gram of material [12g/g]). It is also statistically proven by plotting 95% confidence limit intervals that the difference between these 2 ranges is insignificant. The 425 – 840 micron particle size range gave the next highest absorption of above 10 g/g and the difference was significant in this case. The particles below 106 microns and above 840 microns gave an absorption value of below 6g/g and 7g/g respectively. The variation in 425-840 micron and 38-106 micron particle sizes is due to incorrect samples.

6.2. Effect of Particle Size on Absorption

6.2.1. Proportion of water within and between the particles

Depending on the kind of absorbent, the water holding areas can be divided into two zones,

a) Area between the particles/fibers: The water held between the particles is due to the formation of capillaries, this kind of absorption (most commonly found), is called Capillary absorption. The water held in these capillaries can be removed on the application of very little force.

b) Area within the particles/fibers: Some materials have a very unique structure that has tiny micro-pores in them and they serve as trap zones for fluids. This kind of absorption mechanism is called Micro-porous absorption and to remove the fluids trapped within these pores, a certain amount of force has to be applied.
Core absorbs water both through capillaries and the micro-pores present in their structure. A centrifuge was used to apply a certain amount of force and determine the proportions of water absorbed in the capillary and micro-porous regions.

6.2.1.1. Centrifuge Experiment

3 grams of ground Kenaf Core particles (all size ranges) were soaked in 500 ml water for a period of 30 mins. The water soaked particles were drained using a cheese-cloth. Water was allowed to drain from these particles for a period of up to 10 minutes until the water is completely drained out due to gravity. Care was taken that the particles are not squeezed, to retain the water present within the micro-pores. The particles were then subjected to a centrifuging treatment for a period of 5 minutes to ensure the removal of all the water present in the capillaries. Care was taken to avoid any removal of water trapped within the micro-pores.

**Drawback:** The centrifugal force applied to these particles could not be controlled as the instrument used had a constant speed motor. It is very likely that the force applied was high enough to remove water present in the micro-pores as well.

To overcome this drawback, certain assumptions were made.

a) The force generated by the centrifuge was not high enough to remove the water from within the micro-pores

b) The centrifuging process removed all the water present between the capillaries and retained all the water within in the micro-pores.
The centrifuged particles were immediately tested for amount of water they had retained in the Moisture Analyzer.

Results – Capillary (between particles) vs. Micro-porous (within particles) absorption
Fig. 27

Absorption within Particles

Absorption (gms of water / gm of sample)

Particle Size (um)

Fig. 28

Absorption between particles

Absorption (gms of water / gm of sample)

Particle Size (um)
As shown in the graph, the micro-porous absorption increases gradually with increase in particle size and statistical analysis prove that the increase is insignificant when compared to the total absorption. It can also be said from the comparison between fig. 27 and fig. 28 that as the micro-porous absorption (2 – 4 g/g) has a relatively less effect on the ultimate absorption than the capillary absorption (3 – 10 g/g).

Particles below 38 microns were too small for this experiment. They formed a paste when they were centrifuged and stuck to the cheesecloth. It was very difficult to separate individual particles and test the water present within them.

6.2.2. **Shape and profile of the particles**

Capillary absorption is the mechanism of absorption between 2 or more particles and it directly related to the shape and the profile of the absorbent particles or fibers. It is also determined that in the case of this research, capillary absorption contributes extensively to the most of the absorption taking place. To elaborate on the issue of capillary absorption and to determine the causes for varying absorption levels for different particle sizes, the shape and the profile of the absorbent particles were studied under a Scanning Electron Microscope (SEM).

6.2.2.1. **Scanning Electron Microscope Image Analysis**

A sample of the ground core particles were scattered on a conductive double sided adhesive carbon tape. Particles were scattered on the carbon tape and placed under the SEM. The SEM had a 5KV accelerating voltage, 80 µ aperture and 72 ampere beam current on a
Hitachi S 3200. The working distance was between 15 to 28 mm, the pressure was set to 55 Pa and to avoid charging of electrons 200 Å gold coating was applied.

The first set of images was taken at the lowest magnification, a scale of 1000 microns (1mm) to show the difference between the sizes of various particles from a distance. These images indicate that the difference in particle sizes accompanies a difference in their outer shape as well. The particles below 38 microns (image a) look like tiny spherical specks and as they get bigger they are more easily visible and have a different profile to them (image b, c, d, e and f).
All the particles, irrelevant of their size, have micro-pores in them as shown in the second set of images. The scale of all the pictures shown below is under 20 microns and these images give the details of the micro-pores in every particle size. It is clearly evident from the images that there is a distinct difference between the structure and arrangements of the micro-pores depending on the size of the particle. The pores are perfectly round and well structured when the particle size is large (image d, e and f) but as the particle size gets
smaller (image a, b and c), it is clearly seen that these pores are being ruptured and some of
the pores are opening and breaking apart.

This implies that as the particles become smaller; the pores, instead of serving as trap
zones, just allow the water to pass through them. This visual analysis relates to the results
obtained from the centrifuge experiment done earlier, according to which the micro-porous
absorption decreases with decrease in particle size and vice-versa.

a) Below 38 µ  

b) 38 – 106 µ

c) 106 – 250 µ  

d) 250 – 425 µ
The third sets of images are taken at the scale of the particle size range, i.e. the scale varies depending on the size of the particle. These images are taken to explain the exact shape and the profile of these particles. The particles below a 106 microns (image a and b) have a shape that is close to spherical, the slightly bigger particles between the range of 106 – 425 microns (image c and d) are almost perfect cylinders with imperfect or broken edges and particles bigger than these, above 425 microns (image e and f), range from imperfect cylinders to completely irregular in shape.
c) 106 – 250 µ          d) 250 – 425 µ

e) 425 – 840 µ          f) Above 840 µ

Summarizing the SEM analysis, it can be concluded that there are different shapes for different particle sizes. The ones below 106 microns are roughly spherical in shape, particles between 106 to 425 microns are almost cylindrical in shape and once the particles are above 425 microns, they range from rough cylinders to being irregular in shape.

6.2.2.2. Microscope measurement (Aspect ratio)

Another feature observed during the SEM analysis was the length to diameter ratio (aspect ratio) of the particles. To take the issue further, all the particles were manually
measured under an optical microscope using the Image Analysis Software. The microscope was available in the NCRC Microscopy Laboratory. The magnification was in the range of 2x (above 840 microns) to 25x (38-106 microns). 100 particles were manually measured for every particle size range for length, diameter or width and their aspect ratio.

Results – Aspect Ratio of Untreated Kenaf Core particles

![Aspect Ratio Graph](image)

Fig. 29

The trend obtained by plotting the aspect ratio of various particles matched well with the trend of the absorption within particles (fig. 27). Statistical analysis (scatter plot matrix) revealed that the correlation between the aspect ratio and the capillary absorption is 83%. This indicates that aspect ratio has a remarkable effect on the capillary absorption of the core
particles. Particles below 38 microns required a higher magnification and that could be done only through the SEM, so they were not considered for this analysis.

There might be other factors like surface tension, wettability and the contact angle between the liquid and the absorbent that could affect the capillary absorption but it was considered out of the scope of this research.

6.3. Bonding of Untreated Core Particles

This testing was done before the particles were chemically treated or refined to determine if a handsheet can be made directly from untreated core. The pulp fibers are relatively long (2–4 mm) they have enough bonding points in them to form a smooth handsheet, but in case of this research, granular core particles of sizes below 1mm were used. Due to the short length of the particles and the lack of hydrogen bonding, they spread out randomly on the wire mesh like sand particles and did not form a handsheet. As a 100% core handsheets was not possible, handsheets were made from a 50/50 blend of core and hardwood pulp. The weight of the handsheets was also increased to 3 OD grams (from the usual 1.2 OD grams) to accommodate the required amount of core (at least 50% by weight).

A total of 6 handsheets of 50/50 blend of core particles and the hardwood pulp were made. The results are indicated below,
[Table 5] Handsheets made from Untreated Kenaf Core Particles of all sizes

<table>
<thead>
<tr>
<th>Handsheet #</th>
<th>Blend (Kenaf / Pulp)</th>
<th>Kenaf Core Particle Size (µ)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0 / 100</td>
<td>-</td>
<td>Smooth Handsheet</td>
</tr>
<tr>
<td>2</td>
<td>50 / 50</td>
<td>425 - 840</td>
<td>Rough Handsheet</td>
</tr>
<tr>
<td>3</td>
<td>50 / 50</td>
<td>250 - 425</td>
<td>Rough Handsheet</td>
</tr>
<tr>
<td>4</td>
<td>50 / 50</td>
<td>106 - 250</td>
<td>Rough Handsheet</td>
</tr>
<tr>
<td>5</td>
<td>50 / 50</td>
<td>38 - 106</td>
<td>Smooth Handsheet but loss of Kenaf Core particles</td>
</tr>
<tr>
<td>6</td>
<td>50 / 50</td>
<td>Below 38</td>
<td>Smooth Handsheet but loss of Kenaf Core particles</td>
</tr>
</tbody>
</table>

Weight of handsheets: 3 OD grams
Smooth Handsheet: Perfect blending of fibers into a handsheet
Rough Handsheet: Handsheet obtained with 2 distinct regions a) Pulp base and b) Kenaf particles scattered over the pulp base.
6.4. Effect of Chemical/Water treatment

6.4.1. Absorption test

This graph shown below gives a comparison of the treated highly absorbent samples (water, alkali and cooked) against the untreated core samples. The effect of individual experiments on the absorption properties is also discussed. The results were not analyzed in detail as the experiment was done to determine if there is enhancement in bonding between the core particles without significant loss in absorption.
6.4.1.1. Absorption of water treated particles

It was statistically revealed that in comparison with untreated particles, water treated particles did not show any significant difference in absorption properties. The 106 – 250 micron particles gave a slightly higher absorbency (13 g/g) and this can be due to the removal of dirt and other foreign matter from the particles. The water treated particles also gave a soft feel after they were dried and this can be attributed to the scouring action given to them.
6.4.1.2. Absorption alkali treated particles

![Absorption of Alkali Treated Core](chart.png)

**Fig. 32**

Alkali treated particles gave the third highest absorbency when compared with all other samples (water treated, untreated and cooked). The reason for the decrease in absorption could be that, the treatment with alkali compressed the micro-pores present in the material, restricting the flow of water through them. There was some removal of lignin and waxes due to the alkali action given and it was physically felt in the particles, but as anticipated there was no evident swelling of the particles that might lead to an increase in absorption.
6.4.1.3. Absorption of cooked particles

![Absorption of Cooked Core](image)

Fig. 33

Cooked particles gave the least absorbency. The core particles were treated in the exact same conditions as they would be while making paper. One of the reasons for the drastic decrease in absorbency could be that, when the particles were being cooked at such an elevated temperature, the micro-pores could have been compressed and made it difficult for the liquid to pass into the pores.

The centrifuge experiment done earlier to the untreated particles had revealed that the micro-porous absorption was only up to 2-4 g/g but the difference in absorption levels of
cooked and the untreated particles is more than 4 g/g. This implies that cooking the particles does not only affect the micro-porous absorption but also affects the capillary absorption.

6.4.2. Bonding test

Handsheets were made from all the treated particles to see if there was any enhancement in bonding. A 100% treated core handsheet was still not possible, so a handsheet was made with a 50/50 blend of treated (alkali, cooked and water treated) core particles and hardwood pulp.

Cooked particles gave a comparatively less rough sheet but still showed 2 distinct layers. Alkali treated particles did not show any significant improvement in bonding and water treated particles were as bad as the untreated particles. Physically, there was also change in color of the handsheets obtained. The cooked particles were brown in color, alkali treated particles were yellow in color, water treated particles were relatively whiter than the untreated ones.

[Table 6] Handsheets made from treated core

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Feel</th>
<th>Color</th>
<th>Layers observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>Very rough</td>
<td>Off-White</td>
<td>two distinct layers</td>
</tr>
<tr>
<td>Water treated</td>
<td>Very rough</td>
<td>white</td>
<td>two distinct layers</td>
</tr>
<tr>
<td>Alkali treated</td>
<td>Mildly rough</td>
<td>yellow</td>
<td>two distinct layers</td>
</tr>
<tr>
<td>Cooked</td>
<td>Slightly rough</td>
<td>Brown</td>
<td>two distinct layers</td>
</tr>
</tbody>
</table>

All handsheets obtained showed 2 distinct layers and it was concluded that further enhancement in bonding was required.
6.5. Effect of Refining

In spite of the water/chemical treatment, the particles did not bond well. A 100% core handsheet was not obtained and even a 50/50 blend of core / hardwood pulp produced a rough handsheet with two distinct layers as in the case of handsheets made from untreated particles. To further enhance the bonding, refining was done and after refining, the particles were tested for absorption and bonding properties.

6.5.1. Absorption test

![Absorption of Refined Core](image)

Fig. 34
As shown in the graph, refining does not have a significant effect on the absorption properties of the particles. It can also be seen that, refining of the particles between the size ranges of 106 – 840 scarcely makes a difference to the absorption capacity of the material. Refining also negates the effect caused due to the water treatment given to the particles as there is no specific trend seen between the water treated and untreated samples.

Refining causes fibrillation that increases the binding points in the final handsheet, but also breaks the cell wall, resulting in a reduction in fiber length (aspect ratio) causing a loss in absorption. An optimum refining time can be determined that would give sufficient bonding and a slightly less loss in absorption.

All the samples tested showed absorption of around 9 to 11 g/g irrelevant of the size range or the treatment. The overall values went down due to the fibrillation of the fibers, but the drop in absorption was not significant.

6.5.2. Bonding test

Once the core particles were refined, they were air dried and handsheets were made from them. Although a 100% Core handsheet was not possible, a handsheet with 70/30 blend of core /hardwood pulp was made successfully. These handsheets made from refined particles were smooth and they blend themselves very well with the hardwood pulp giving a handsheet that had a single layer. The core/pulp handsheet was a little stiff in comparison with the handsheet made from a 100% hardwood pulp and this was due to the short length of the core fibers (0.6mm). SEM pictures of the refined particles were taken to visually understand the effect of refining on the Core particles.
SEM Analysis

The SEM had a 5KV accelerating voltage, 80 μ aperture and 72 ampere beam current on an S 3200. The working distance was between 15 to 28 mm, the pressure was set to 55 Pa and to avoid charging of electrons 200 Å gold coating was applied. The images shown below are of 106-250 Microns (Image a, b and c), 250-425 microns (Image d, e and f) and 425-840 Microns (Image g and h).
6.6. Handsheets from Refined Core Particles

Once these particles were tested for absorption, handsheets were made from them. The core content every handsheet made varied from 50–70% by weight, depending on the particle size, the rest was hardwood pulp. The core and pulp proportions were weighed and passed through a pulp disintegrator for 400 cycles and were then put into the handsheet maker.
The handsheets made were compared to fluff pulp, the leading commercial product that is used as the absorbent core. Fluff pulp samples were obtained from Buckeye Technologies Inc. The pulp was obtained in a sheet form, which was disintegrated into fibrous matter and then the fibers were fluffed. The fluffed fibers were centrifuged and dried for a couple of days. 100% Fluff pulp handsheets were made of the same basis weight.

6.6.1 Absorption Test

The following 8 batches of core/hardwood pulp handsheets were obtained and tested in GATS for absorption and compared to handsheets made of 100% fluff pulp. Due to lack of samples only 2 tests were carried out for each handsheet.

a) 106 – 250 microns Untreated Un-Ref  
b) 106 – 250 microns Water Treated Un-Ref  
c) 106 – 250 microns Untreated refined  
d) 106 – 250 microns Water treated refined  
e) 250 – 425 microns untreated refined  
f) 250 – 425 microns water treated refined  
g) 425 – 840 microns untreated refined  
h) 425 – 840 microns water treated refined
As seen in the absorption of refined particles (fig. 34), there is no difference when the particle size range is between 106 – 840 microns. Once the particles are refined the difference in absorption levels no longer exists and all the particles give the same range of absorption. In case of handsheets, the absorption is lower than particles because of the presence of hardwood pulp in the sheet. The pulp absorbs lesser amount of water when compared to the core, but it is necessary to serve as a base to get the handsheet off the mesh of the handsheet maker.

The rough handsheets made from un-refined 106-250 particles gave a higher absorption (above 8.5 g/g) when compared to the rest (around 6.5 – 7 g/g). This data reveals
that refining has a negative effect on the absorption properties of the core particles. Handsheets from un-refined particles above 250 microns were tried, but they gave a rough feel to the handsheet and the particles were scattered on the surface giving two distinct regions. Particles in the range of 106 – 250 microns also gave a rough feel in comparison with the refined handsheets but they had bonded well enough to the pulp sheet and it was easy to get them off the mesh of the handsheet maker.
Chapter 7 – Manufacturing of an Absorbent

Nonwoven

After all the chemical and refining experiments were carried out, 7 different samples were chosen to make the final handsheets that were to be incorporated in between the spun-bond webs. The particles that were chosen to make these handsheets were the untreated and water treated 106 – 250, 250 – 425, 425 – 840 micron particles, as these were the ones that gave the highest degree of absorbency. All these particles were subjected to refining treatment in the blender for an hour. There was also a batch of unrefined handsheets made from 106 – 250 microns particles. This was due to the fact that they gave an adequate blending and the handsheet did not show up as two distinct layers, but still the handsheet obtained was rough. It was made sure that all the handsheets have a minimum of 50% by weight of Kenaf Core.

The Kenaf/pulp handsheets weighed around 160 grams / Sq. meter and the diameter of the handsheet was 6”. A total of 15 batches of handsheets were prepared from different samples, this also included 2 batches of handsheets made from fluff pulp for comparison. Each of these batches was sandwiched between a pair of spun-bond webs. The spun-bond fabric used was a 17 grams / Sq. meter polypropylene web that was lightly calendar bonded.
The spun-bond fabric was roughly 20” wide and depending on the availability of handsheets, they were placed in 2 or 3 rows in between the spun-bond fabrics. The fabrics with the handsheets between then were passed through the hydro-entanglement for bonding. The pressures used in the hydro-entangled jets were 40 bar, 75 bar and 120 bar in the first three manifolds respectively. After the bonding, the fabric was passed through a dryer at a drying temperature of 100°C. The fabric was processed at a speed of 10 m/min. The forming wire used was 100 mesh and the belt used was formtec 103. The samples once passed through the dryer were wound onto a roller and the samples were obtained.

The ratio of the spun-bond webs and the absorptive core was supposed to be 50/50 but producing high concentration Kenaf Core handsheets with lower basis weight had a lot of complications. One of the reasons for producing the handsheet with a high basis weight was to get the required amount (at least 50% by weight) of kenaf core in the handsheet. Handsheets with lower basis weight and high kenaf core content were tried, but a reduction in pulp below 50 grams / sq. meter gave insufficient bonding and the handsheet did not come off the mesh of the handsheet maker. The kenaf handsheets made required a pulp base over which they could deposit themselves and act as the absorptive material. Thus to increase the kenaf content in the handsheets above 50%, heavier handsheets (up to 160 grams / sq. meter) were made.
Chapter 8 – Conclusions

- The optimum Kenaf Core particle size range that gives maximum water absorbency is between 106 – 425 microns.
- Kenaf Core absorbs water due to capillaries and micro-pores but capillary absorption is significantly higher and plays a major role.
- The Aspect Ratio (Length : Diameter) perfectly co-relates with the capillary absorption.
- Alkali treatment of Core particles causes a slight reduction in absorption and cooking them under high temperature causes a drastic reduction in absorption.
- Refining of Core particles causes a slight reduction in absorption due to fibrillation.
- Handsheets with a blend of 70/30 (Core / hardwood pulp) can be made and a reduction in hardwood pulp below 30% causes instability and the handsheet does not come off the mesh of the handsheet maker.
Chapter 9 – Recommendations

- Enhance the absorption of Kenaf Core particles by chemical treatment.
- Determining the optimum refining treatment to prevent the loss in absorbency.
- To make handsheets with a blend of fluff pulp and Kenaf Core.
List of References


3. LaGreta et al 1994


List of Tables


4) Experiments conducted in this research

5) Results obtained in this research

6) Results obtained in this research

7) Results obtained in this research [Appendix]

8) Results obtained in this research [Appendix]

9) Results obtained in this research [Appendix]

10) Results obtained in this research [Appendix]

11) Results obtained in this research [Appendix]

12) Results obtained in this research [Appendix]

13) Results obtained in this research [Appendix]

14) Results obtained in this research [Appendix]
## APPENDIX

[Table 7] Absorption of untreated core particles

<table>
<thead>
<tr>
<th>Time</th>
<th>Below 38</th>
<th>38-106</th>
<th>106-250</th>
<th>250-425</th>
<th>425-840</th>
<th>Above 840</th>
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<tr>
<td>0</td>
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<td>200</td>
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<td>6.82</td>
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<td>300</td>
<td>3.21</td>
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<td>10.02</td>
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<td>400</td>
<td>3.27</td>
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<td>8.94</td>
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<td>500</td>
<td>3.32</td>
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<td>600</td>
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<td>3.44</td>
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<td>3.51</td>
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<td>12.00</td>
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<td>1500</td>
<td>3.58</td>
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<td>12.18</td>
<td>11.88</td>
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<td>1600</td>
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<td>11.94</td>
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<td>1700</td>
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<td>12.30</td>
<td>12.00</td>
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<td>Mean Absorption (g/g) @ 1800 secs</td>
<td>3.65</td>
<td>5.51</td>
<td>12.36</td>
<td>12.06</td>
<td>10.53</td>
<td>6.79</td>
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<td>St Dev @ 1800 secs</td>
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<td>1.81</td>
<td>0.79</td>
<td>0.55</td>
<td>1.31</td>
<td>0.21</td>
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<td>Lower 95% @ 1800 secs</td>
<td>3.31</td>
<td>3.26</td>
<td>11.64</td>
<td>11.55</td>
<td>8.89</td>
<td>6.53</td>
</tr>
<tr>
<td>Upper 95% @ 1800 secs</td>
<td>3.99</td>
<td>7.75</td>
<td>13.09</td>
<td>12.57</td>
<td>12.16</td>
<td>7.06</td>
</tr>
<tr>
<td>Std. Error @ 1800 secs</td>
<td>0.12</td>
<td>0.81</td>
<td>0.30</td>
<td>0.21</td>
<td>0.59</td>
<td>0.10</td>
</tr>
</tbody>
</table>
### [Table 8] Centrifuge Experiment – Proportion of water held within vs between particles

<table>
<thead>
<tr>
<th>Particle Size (um)</th>
<th>Moisture Analyzer Reading (%)</th>
<th>Std. Error</th>
<th>St. Dev</th>
<th>Water retained within pores after centrifuging (g/g of material) [M.A]</th>
<th>Total absorption</th>
<th>Water between particles (g/g of material) [C.A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Below 38</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>3.65</td>
<td>-</td>
</tr>
<tr>
<td>38 – 106</td>
<td>41.24</td>
<td>0.03</td>
<td>0.05</td>
<td>2.43</td>
<td>5.51</td>
<td>3.08</td>
</tr>
<tr>
<td>106 – 250</td>
<td>37.60</td>
<td>0.04</td>
<td>0.06</td>
<td>2.66</td>
<td>12.36</td>
<td>9.70</td>
</tr>
<tr>
<td>250 – 425</td>
<td>35.13</td>
<td>0.09</td>
<td>0.13</td>
<td>2.85</td>
<td>12.06</td>
<td>9.21</td>
</tr>
<tr>
<td>425 – 840</td>
<td>30.37</td>
<td>0.09</td>
<td>0.13</td>
<td>3.29</td>
<td>10.53</td>
<td>7.24</td>
</tr>
<tr>
<td>Above 840</td>
<td>28.57</td>
<td>0.10</td>
<td>0.14</td>
<td>3.50</td>
<td>6.78</td>
<td>3.28</td>
</tr>
</tbody>
</table>

### [Table 9] Aspect Ratio

<table>
<thead>
<tr>
<th>Particle Size (um)</th>
<th>Length</th>
<th>Width</th>
<th>Aspect Ratio</th>
<th>S.D (2σ)</th>
<th>S.E.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Below 38</td>
<td>-</td>
<td>-</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>38 - 106</td>
<td>49.71</td>
<td>28.72</td>
<td>1.99</td>
<td>1.66</td>
<td>0.30</td>
</tr>
<tr>
<td>106 - 250</td>
<td>557.09</td>
<td>144.41</td>
<td>3.80</td>
<td>2.06</td>
<td>0.38</td>
</tr>
<tr>
<td>250 - 425</td>
<td>797.68</td>
<td>279.14</td>
<td>2.85</td>
<td>1.9</td>
<td>0.35</td>
</tr>
<tr>
<td>425 - 840</td>
<td>1044.85</td>
<td>487.42</td>
<td>2.22</td>
<td>1.28</td>
<td>0.23</td>
</tr>
<tr>
<td>Above 840</td>
<td>1743.00</td>
<td>840.42</td>
<td>2.20</td>
<td>1.6</td>
<td>0.29</td>
</tr>
</tbody>
</table>
**[Table 10] Absorption of water treated particles**

<table>
<thead>
<tr>
<th>Time (secs)</th>
<th>106 - 250</th>
<th>250 - 425</th>
<th>425 - 840</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>100</td>
<td>7.81</td>
<td>5.31</td>
<td>4.62</td>
</tr>
<tr>
<td>200</td>
<td>11.63</td>
<td>8.42</td>
<td>6.91</td>
</tr>
<tr>
<td>300</td>
<td>12.85</td>
<td>10.06</td>
<td>8.31</td>
</tr>
<tr>
<td>400</td>
<td>13.36</td>
<td>10.93</td>
<td>9.11</td>
</tr>
<tr>
<td>500</td>
<td>13.58</td>
<td>11.32</td>
<td>9.50</td>
</tr>
<tr>
<td>Mean absorption (g/g) @ 600 secs</td>
<td>13.67</td>
<td>11.52</td>
<td>9.70</td>
</tr>
<tr>
<td>St. Dev @ 600 secs</td>
<td>1.10</td>
<td>1.17</td>
<td>1.31</td>
</tr>
<tr>
<td>Lower 95% @ 600 secs</td>
<td>12.30</td>
<td>10.42</td>
<td>7.59</td>
</tr>
<tr>
<td>Upper 95 @ 600 secs</td>
<td>15.03</td>
<td>12.6</td>
<td>11.79</td>
</tr>
<tr>
<td>Std. Error @ 600 secs</td>
<td>0.49</td>
<td>0.44</td>
<td>0.65</td>
</tr>
</tbody>
</table>

**[Table 11] Absorption of alkali treated particles**

<table>
<thead>
<tr>
<th>Time</th>
<th>106 - 250</th>
<th>250 - 425</th>
<th>425 - 840</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>100</td>
<td>6.63</td>
<td>4.48</td>
<td>3.94</td>
</tr>
<tr>
<td>200</td>
<td>9.16</td>
<td>7.46</td>
<td>6.18</td>
</tr>
<tr>
<td>300</td>
<td>10.14</td>
<td>8.70</td>
<td>7.36</td>
</tr>
<tr>
<td>400</td>
<td>10.64</td>
<td>9.06</td>
<td>7.82</td>
</tr>
<tr>
<td>500</td>
<td>10.95</td>
<td>9.22</td>
<td>8.00</td>
</tr>
<tr>
<td>Mean absorption (g/g) @ 600 secs</td>
<td>11.15</td>
<td>9.32</td>
<td>8.10</td>
</tr>
<tr>
<td>St. Dev @ 600 secs</td>
<td>0.44</td>
<td>0.5</td>
<td>2.12</td>
</tr>
<tr>
<td>Lower 95% @ 600 secs</td>
<td>10.45</td>
<td>9</td>
<td>6.25</td>
</tr>
<tr>
<td>Upper 95 @ 600 secs</td>
<td>11.85</td>
<td>9.64</td>
<td>10.39</td>
</tr>
<tr>
<td>Std. Error @ 600 secs</td>
<td>0.40</td>
<td>0.26</td>
<td>0.83</td>
</tr>
</tbody>
</table>
### Table 12: Absorption of cooked particles

<table>
<thead>
<tr>
<th>Particle Size (microns)</th>
<th>Time</th>
<th>106 - 250</th>
<th>250 - 425</th>
<th>425 - 840</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>2.43</td>
<td>1.41</td>
<td>1.85</td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>4.28</td>
<td>2.59</td>
<td>3.18</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>5.78</td>
<td>3.65</td>
<td>4.32</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td>6.97</td>
<td>4.60</td>
<td>5.31</td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>7.79</td>
<td>5.46</td>
<td>6.07</td>
<td></td>
</tr>
<tr>
<td>Mean absorption (g/g) @ 600 secs</td>
<td>8.32</td>
<td>6.22</td>
<td>6.62</td>
<td></td>
</tr>
<tr>
<td>St. Dev @ 600 secs</td>
<td>0.29</td>
<td>0.46</td>
<td>0.35</td>
<td></td>
</tr>
<tr>
<td>Lower 95% @ 600 secs</td>
<td>7.63</td>
<td>5.07</td>
<td>5.82</td>
<td></td>
</tr>
<tr>
<td>Upper 95 % @ 600 secs</td>
<td>9.05</td>
<td>7.37</td>
<td>7.55</td>
<td></td>
</tr>
<tr>
<td>Std. Error @ 600 secs</td>
<td>0.16</td>
<td>0.27</td>
<td>0.2</td>
<td></td>
</tr>
</tbody>
</table>

### Table 13: Absorption of refined core particles

<table>
<thead>
<tr>
<th>Particle Size (microns)</th>
<th>untreated</th>
<th>water treated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean absorption (g/g) @ 600 secs</td>
<td>10.54</td>
<td>10.25</td>
</tr>
<tr>
<td>St. Dev @ 600 secs</td>
<td>0.27</td>
<td>1.05</td>
</tr>
<tr>
<td>Lower 95% @ 600 secs</td>
<td>9.88</td>
<td>7.65</td>
</tr>
<tr>
<td>Upper 95 @ 600 secs</td>
<td>11.21</td>
<td>12.86</td>
</tr>
<tr>
<td>Std. Error @ 600 secs</td>
<td>0.15</td>
<td>0.61</td>
</tr>
</tbody>
</table>
## Table 14: Absorption of Handsheets

<table>
<thead>
<tr>
<th>Particle Size (microns)</th>
<th>Treatment</th>
<th>Absorption (g/g)</th>
<th>St Error</th>
<th>St Dev</th>
</tr>
</thead>
<tbody>
<tr>
<td>106-250 Un-refined</td>
<td>Untreated</td>
<td>8.76</td>
<td>0.89</td>
<td>1.26</td>
</tr>
<tr>
<td>106-250 Un-refined</td>
<td>Water Treated</td>
<td>9.15</td>
<td>1.58</td>
<td>2.24</td>
</tr>
<tr>
<td>106-250 Refined</td>
<td>Untreated</td>
<td>6.95</td>
<td>1.38</td>
<td>1.96</td>
</tr>
<tr>
<td>106-250 Refined</td>
<td>Water Treated</td>
<td>7.16</td>
<td>0.55</td>
<td>0.78</td>
</tr>
<tr>
<td>250-425 Refined</td>
<td>Untreated</td>
<td>6.86</td>
<td>0.65</td>
<td>0.92</td>
</tr>
<tr>
<td>250-425 Refined</td>
<td>Water Treated</td>
<td>6.57</td>
<td>0.76</td>
<td>1.08</td>
</tr>
<tr>
<td>425-840 Refined</td>
<td>Untreated</td>
<td>6.69</td>
<td>1.52</td>
<td>2.16</td>
</tr>
<tr>
<td>425-840 Refined</td>
<td>Water Treated</td>
<td>6.94</td>
<td>0.52</td>
<td>0.74</td>
</tr>
<tr>
<td>Fluff pulp 1</td>
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<td>6.89</td>
<td>0.38</td>
<td>0.54</td>
</tr>
<tr>
<td>Fluff Pulp 2</td>
<td></td>
<td>6.99</td>
<td>0.44</td>
<td>0.62</td>
</tr>
</tbody>
</table>