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Effect of alkali treatment on single species wood pulp fiber properties for the application of nursing pad absorbent core

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The present work aims to study the suitability of single species soft wood kraft pulp for the application of absorbent core of the nursing pad. Nursing pads are used inside the brassiere by the lactating mother to absorb the milk leaked from the breast, thereby avoiding the staining of the cloth and a possible embrassment. A new approach for preparing the single species pulp specifically for the production of wet-laid non-woven has been carried out. Alkali treatment at different concentrations ranging from 3% to 18% (w/v) NaOH was carried out to study its influence on the softness of the web produced. The NaOH concentration was optimized with 15% (w/v) based on the physical assessment of softness and flexibility. The properties of 15% NaOH treated and untreated wood pulp was compared. The NaOH treatment improved the absorbency behavior from 12.3 g/g to 15.5 g/g due to the removal of hemicellulose, extractive content, and lignin. The results revealed the hemicellulose content of the untreated and treated pulp as 5.49% and 0.10% respectively. The extractive content of the untreated sample was 0.1% and the treated wood pulp was 0.04% and the kappa number was reduced from 1.409 to 0.93984. The fiber morphology of treated and untreated wood pulp was analyzed using fiber analyzer Kajaani FS 300 and the result revealed the changes in the fiber length, fiber width, fiber coarseness, fiber curl, and fiber kink, etc. These changes provide the suitability of the pulp for absorbent core production for the disposable nursing pad.

Keywords: Absorbent core, alkali treatment, fiber characterization, nursing pad.

Introduction

Nursing pads are used by lactating mothers to prevent staining of cloth and a possible embrassment caused by the milk discharge during and after feeding[1–3]. The general construction of the nursing pad consists of an innermost layer, an absorbent core, and the outer most layer[4]. The main function of the absorbent core is to absorb and retain the liquid. The properties required for the absorbent core of the nursing pad are wettability, absorbency, softness, smoothness, flexibility and conformity to the shape of the breast. The commercially available nursing pad consists of wood pulp, superabsorbent polymer, and synthetic fibers produced by air-laid nonwoven technique. The drawback in the commercially available air-laid nonwoven absorbent core is structural integrity during usage condition[5]. Crow *et al*. (2003), state that alkali treated wet-laid pulp is an excellent material for absorbent core and the samples produced exhibit excellent absorbency and pad integrity[6,7].

Among the cellulosic fibers, wood pulp is used mainly in nonwovens designed for absorbency and porosity[8]. Both hardwood and softwood are used for absorbent core production in commercial products such as disposable diapers, sanitary napkins, adult diapers, and related products[9]. Due to the large difference between softwood and hardwood fibers and also large variations in the different species of wood, the raw material selection plays a major role in this process[10,11]. Preliminary work of wet-laid web preparation has been carried out by procuring softwood pulp. The wet-laid web produced was hard in nature and board-like. Since it could not be used for absorbent core purpose, the necessity for alkali treatment for the procured pulp is observed. Since each pulp has its own chemical composition, NaOH concentration, time, the temperature has to be optimized to ensure maximization of their use in the application as an absorbent core for the nursing pad[12,13].

The chemical composition of the raw wood pulp and bleached kraft wood pulp[8,14] as seen in literature are tabulated in Table 1. The alkali treatment removes the hemicellulose, lignin, and extractives, thereby improving its softness, flexibility, and absorbency, it also facilitates higher brightness

Table 1. Chemical composition of the raw wood pulp and bleached Kraft wood pulp

Chemical composition	Raw wood pulp (%)	Bleached Kraft wood pulp (%)
Cellulose	39	35
Hemicellulose	29	9
Lignin	27	3
Extract	4	0.50

Source: LC Vander Wielen, 2004.

and brightness stability[15]. Literature has references to alkali treatment being carried out for wood pulp samples from 2 to 18%[16–18]. Lund *et al*. carried out alkali treatment at three different concentrations (2%, 4%, and 8%) for softwood pulp and the fluff pad was produced by air-laid nonwoven technique. Fluff pad made from 8% NaOH treated pulp showed better flexibility than networks of untreated softwood pulp and birch pulps[19] due to the changes in fiber morphology[20,21].

The present study, aims to analyse the suitability of single-species softwood kraft pulp for application of absorbent core of the nursing pad. Alkali treatment at different NaOH concentrations ranging from 3% to 18% (w/v) at an interval of 3% was carried out. This influences the change in the fiber morphology and chemical composition of the fiber which has been characterized. Fiber identification was carried out using the microscopic technique. Ash content and moisture content are examined for the pulp procured before alkali treatment for a quality check for its suitability of absorbent purpose. The hemicellulose content, extractive content, lignin content, and total absorption capacity are investigated for comparison of both untreated and 15% alkali treated wood pulp. Analysis of fiber morphology has been carried out by using Kajaani FS 300 fiber analyzer for a comparison of both untreated and 15% alkali treated wood pulp.

Materials and methods:

NaOH reagent (Central Drug House (P.) Ltd., Delhi) was used for the NaOH treatment. Distilled water was used for all the experiments. All other AR grade chemicals used in the experimental studies are supplied by SRL Pvt. Ltd., Delhi, India. Remi overhead stirrer RQ-122, Remi Electrotechnik Ltd., Mumbai, India. Weighing balance model BSA2245-CW, Sartorius were used. The methodology for research is explained through the flow chart as given in Fig. 1.

Selection and identification of wood pulp:

Fully bleached softwood kraft wood pulp was procured from Rajarajeswari Traders, Chennai, India. The pulp was procured in sheet form. Microscopic analysis was done at Tamilnadu Newsprint and Paper Limited (India), Karur, Tamilnadu using a Leica Qwin microscope at a magnification of 100X. The analysis was carried out according to the TAPPI Standard T263 sp-11[22].

Quality check of the procured pulp:

Ash content and moisture content determination of pulp: Ash content and moisture content of the procured pulp was determined by IS 1060 (Part-1), 1966, RA 1992. In ash content test, a sample of known weight was placed in a furnace at a temperature of 900°C and fully combusted. The weight of the residual ash after combustion is reported as the ash content[23]. In moisture content test, a sample of known weight was placed in an oven at a temperature of 105°C till the constant weight of the sample is obtained. The moisture content was determined using the formula given below.

$$\text{Moisture content \%} = \frac{\text{Intial weight of pulp} - \text{Oven dry weight of pulp}}{\text{Oven dry weight of pulp}} \times 100 \quad (1)$$

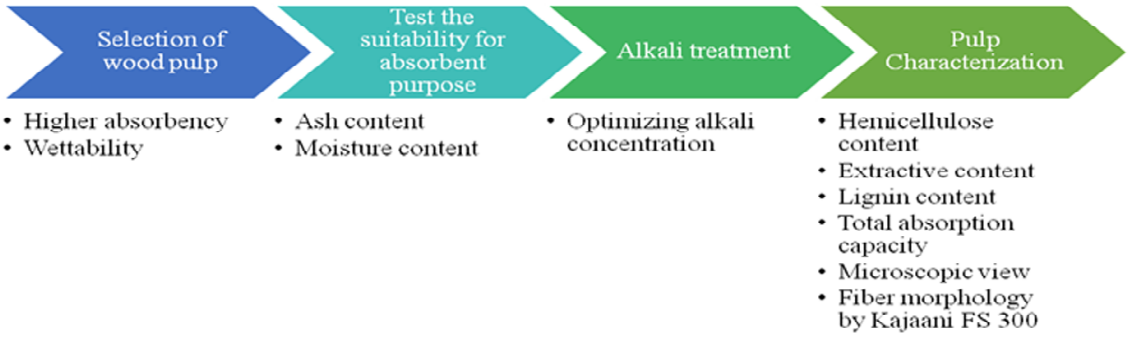


Fig. 1. Methodology.

NaOH treatment:

The pulp was given NaOH treatment using 15% (w/v) NaOH at 32°C (room temperature) for 10 min maintaining material:liquor ratio of 1:33. The measured quantity of pulp sheet obtained was soaked in distilled water for 12 h and the disintegration of the pulp was carried out in the overhead stirrer at 3000 rpm for 10 min. Water was removed from the pulp after filtering the pulp through the sieve of ASTM mesh size 200, in order to avoid wastage of the fiber during filtering. The excess water was squeezed out. The disintegrated pulp was added to the NaOH solution. The thorough stirring of the pulp was carried out for 10 min at 32°C for effective treatment. After treatment,the NaOH solution was removed by thoroughly washing several times using distilled water. The residual NaOH solution was removed by treating in 0.1% (w/v) sulphuric acid solution. It was rinsed in distilled water until neutral in pH. Drying was carried out in a hot air oven at 80°C till a constant weight was obtained. Conditioning of the pulp was carried out in an environment chamber at 25±2°C and 65±2% R.H.

Hemicellulose content of the treated and untreated pulp:

The hemicellulose content was calculated as given in the formula (2). The experiment was carried out as per the Tappi standard T223-Cm84/Um 236[24].

Hemicellulose content % =

$$\left[\frac{7.5\,(V_2 - V_1)N}{W} - 1\right] \qquad (2)$$

where, V_1 = test sample reading, V_2 = blank reading, W = sample weight, N = normality of sodiumthio-sulphate solution, 1.0 = correction factor for other carbohydrates 4 and 3 carbons.

Extractive content of the untreated and treated pulp:

The extractive content of the untreated and treated pulp was determined using the Tappi method T204 cm97 and dichloromethane as a solvent[25]. Then the extractive content was calculated as given in below formula (3).

$$\text{Extractive content \%} = [(W_e - W_b)/W_p] \times 100 \qquad (3)$$

where W_e = oven-dry weight of extract, g, W_p = oven-dry weight of pulp, g, W_b = oven-dry weight of blank residue, g.

Lignin content estimation of untreated and treated pulp:

Kappa number of the untreated and treated pulp was determined according to the Tappi standard T236 om-99[26]. The Kappa number was calculated using the below formula (4).

$$K = \frac{p \times f}{w} \text{ and } P = \frac{(b - a)N}{0.1} \qquad (4)$$

where K = kappa number, f = factor for correction to a 50% permanganate consumption, dependent on the value of p, w = weight of moisture-free pulp in the specimen, g, p = amount of 0.1 N permanganate actually consumed by the test specimen, mL, b = amount of the thiosulfate consumed in the blank determination, mL, a = amount of thiosulfate consumed by the test specimen, mL, N = normality of the thiosulfate.

Total absorption capacity of untreated and treated pulp:

The test was done according to the standard NWSP 010.1.RO. The absorptive capacity of the untreated and treated samples were determined by taking 1 g of the sample and immersing it into distilled water for one minute and made to hang for 2 min to drain completely. The wet weight was taken. The total absorption capacity was determined using the formula given below. It is indicated in g/g of liquid absorbed (5).

Total weight (g) of water absorbed (g/g) =

Wet weight (g) – dry weight (g) (5)

Fiber morphology analysis of untreated and treated pulp:

Fiber morphology was measured with a Kajaani FS-300 (Metso), and results reported were according to TAPPI T271. The test was conducted at TNPL, Karur. The sample was diluted and dispersed before the measurement. 0.02 mm was the smallest fiber length the Kajaani FS-300 could measure. Consistency of the sample was checked after the disintegration. It was adjusted to 0.01% and 50 mL of the sample was taken for testing. Kajaani FS-300 is a class 1 laser device. The analyzer contained a class 3B laser light source. The power of the laser light was 7.5 mW. The instrument consists of a sample inlet capillary tube through which the solution was drawn inside and detected by a laser light source. It had a LED measurement light and a CCD measurement camera. After the measurement, the sample was drawn outside through the outlet. The sample holder held the container with the sample. It had provisions to hold five samples at a time. The measurement was done one after another when programmed.

Results and discussion

Selection and identification of wood pulp fiber:

Fiber length is the main criterion used for the selection of wood pulp. Fully bleached softwood kraft pulp was procured for the following reasons. Softwood fibers are generally 3 to 4 mm in length and 0.035 to 0.045 mm in width. Hardwood fibers are smaller 0.8 to 1.3 mm in length and 0.02 to 0.03 mm in width[27]. Long fibers provide greater structural strength than short fibers due to the greater number of fiber contacts and continuity in the structure. Absorption capacity, however, was higher for longer fiber samples because of greater pad rigidity resulting from longer fibers. The extractable content of kraft pulps being significantly lower than sulphite or mechanical pulps. Based on tests of commercial pulps, it appears that Southern pine kraft pulps are significantly lower in extractable than the pulps made from other species in North America. It has lowest initial contact angle[28].

Since the fiber strength and physical properties are species dependent, knowledge of the species is important for predicting the ultimate strength of the product produced. The fiber species of bleached kraft softwood pulp was examined under microscope. The presence of tracheids confirms the fiber present as softwood fiber as shown in Fig. 2. A comparison of microscopic picture with the reference picture at TNPL library shows the species as pine fiber. Pine is a softwood that produces long, thick and strong fibers. These qualities contribute to the strength of the absorbent core.

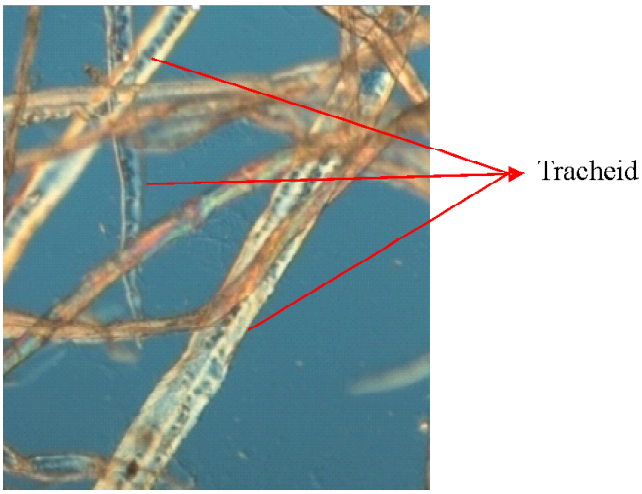


Fig. 2. Microscopic image of softwood Kraft pulp.

Ash content determination of pulp:

The ash content test result which was 0.03%, showed the pulp procured having less contaminents like mineral matter, metallic matter and other additives such as filling, coating, pigmenting or other added materials and therefore can be used for the absorbent core of the nursing pad[8].

Moisture content determination of pulp:

The moisture content test results obtained for the procured pulp was 13.39%. Wood pulp is a hygroscopic material when fully dried it absorbs water vapor from the atmosphere. The moisture content of the pulp depends on material composition factors such as fiber species and additives added. Climatic conditions during collection, transport, baling, and storage of the pulp (outdoors, undercover, or in enclosed facilities) also have an effect. The extra moisture on the surface of the fibers promotes bonding of the fibers when they come in contact with each other. Therefore the moisture content of the pulp should be at the right target. Moisture content of 6–10% was maintained in the production of commercial pulp sheets[14]. Hence moisture content result proves its suitability for further processing in wet condition.

Effect of alkali treatment on wood pulp:

Alkali treatment was carried out at different NaOH concentrations ranging from 3% to 18% (w/v) at an intervals of 3% w/v to improve the softness of the wood pulp. Up to 12% (w/v) alkali concentration it was observed that the hardness of the pulp doesn't change. Only at 15% (w/v) alkali concentration the softness of the pulp is achieved. Further increase in alkali concentration to 18% (w/v) drastic weight loss of fiber was observed. Hence the NaOH concentration of 15% (w/v) has been optimized. Fig. 3 shows the appearance of (a) untreated wet-laid web and (b) 15% NaOH treated wet-laid web sample.

During alkali treatment the OH groups present in the wood pulp (cellulose I) reacts with NaOH to form alkali cellulose[13]. When this alkali is washed, a transformation into cellulose II occurs as given in the Fig. 4.

Fig. 5 shows the mechanism of NaOH treatment with wood pulp. Changes in the morphology of the cellulosic materials due to the swelling occurs; swelling can be either interfibrillar or intrafibrillar in character or both depending on

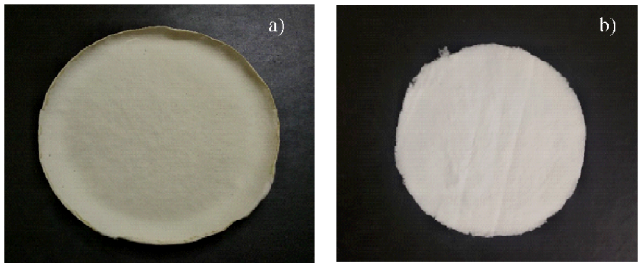


Fig. 3. Appearance of (a) untreated wet-laid web; (b) 15% NaOH treated wet-laid web.

ing the removal of hemicelluloses[33]. The NaOH treatment caused an overall decrease in the degree of crystallinity[34–36]. Fig. 6 shows the microscopic image of untreated fiber (a) which has flat ribbon like structure and NaOH treated pulp fiber (b) which shows round shaped smooth structure reveals the swelling of fibers takes place after NaOH treatment.

Characterization of alkali treated wood pulp samples:

The results of the characterization evaluation was summarized in the Table 2.

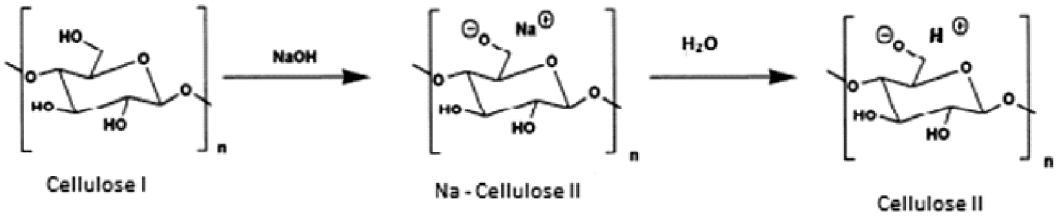


Fig. 4. Chemical reaction of alkali treatment with wood pulp.

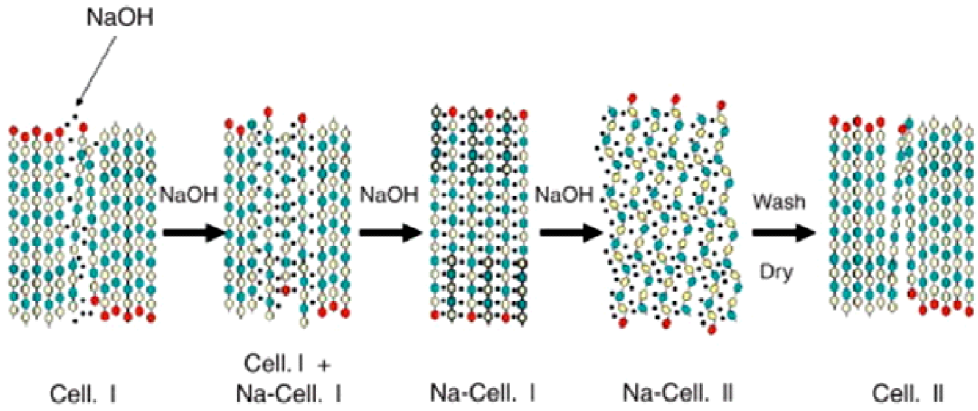
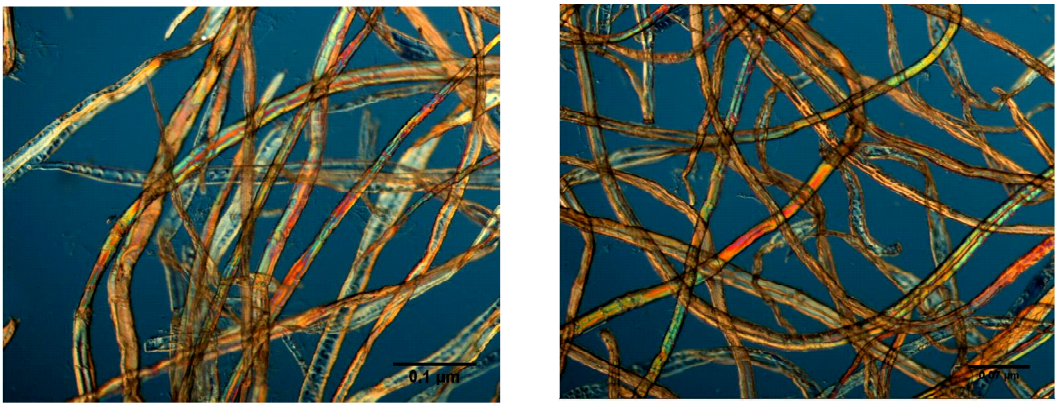


Fig. 5. Possible mechanism of alkali treatment with wood pulp (Source: Okano and Sarko, 1985).

the concentration of the NaOH solution. At higher concentration Na^+ cations can efficiently diffuse into the crystalline structure of the native cellulose. The cellulose undergo the lattice transformation from parallel arrangement (cellulose I) due to the diffusion of NaOH both in the amorphous and crystalline region to the anti-parallel arrangement (cellulose II) crystal structure when the alkali was removed by washing with water[29–32]. The fiber wall gets swollen due to osmotic effects and the swelling pressure disrupted the hydrogen bonds between the fibrils building up the fiber wall thereby facilitat-

Hemicellulose determination of wood pulp:

Test results revealed reduction in hemicellulose content from 5.49 to 0.10% for untreated and NaOH treated pulp respectively. This shows the maximum removal of hemicellulose content from the procured wood pulp which will help improvement to the absorbency. The fiber swelling during alkali treatment is responsible for the removal of hemicellulose from the pulp fiber, this creates the morphological and chemical composition changes in the wood pulp[33].



(a) (b)

Fig. 6. Microscopic image of wood pulp fiber (a) untreated fiber; (b) 15% NaOH treated fiber.

Table 2. Summary of characterization evaluation results

Soft wood Kraft pulp	Hemicellulose (%)	Extractive content (%)	Lignin content (Kappa number)	Total absorption capacity (g/g)
Untreated	5.49	0.1	1.409	12.3
15% NaOH treated	0.10	0.04	0.939	15.5

The middle lamella and primary wall are mostly non-cellulosic whereas the chief chemical components of the secondary walls are cellulose and hemicellulose. The hemicellulose may lie between the cellulosic microfibrils either as an amorphous or as a crystalline granular material, or may form their own microfibrils which may or may not have crystalline regions. Therefore, the mechanism of removal of hemicellulose involves both chemical and physical phenomena[17].

Extractive content of wood pulp:

Extractive content was tested and found for untreated wood pulp was 0.1% which was reduced to 0.04% after 15% NaOH treatment. Extractive content is one of the most important factors in the wicking rate. The relation between the wicking rate of various fluff and the extractive content shows a significant correlation in the depression of wicking rate by increased extractives[21]. Even if the extractive content is 0.28% the wicking rate (cm/sec) is 0.14 and, when it is reduced to 0.02%, the wicking rate (cm/sec) has been increases to 0.44. So it is necessary to remove the extractives to the possible extent.

Lignin content of wood pulp:

Kraft and bleaching process would remove the lignin content to a greater extent and when tested was found to have 1.409 Kappa number. Further, this caused a reduction to 0.939 Kappa number after 15% NaOH treatment.

Total absorbency capacity of wood pulp:

Total absorbency capacity was found to be improved from 12.3 g/g (untreated pulp) to 15.5 g/g after 15% NaOH treatment. This result was also correlated with the removal of the extractive content.

Fiber morphology:

The fiber properties and fiber morphology such as fiber length, width, coarseness, curl, and kink, undergo changes during alkali treatment. These changes can have a marked effect on the quality and end-use performance of the products. Hence the fiber morphology is analysed after NaOH treatment and compared with untreated wood pulp using Kajaani FS-300 fiber analyzer. The results are tabulated in Table 3.

Fiber length:

During NaOH treatment, swelling of the fibers takes place leading to the reduction of fiber length. Average fiber length is an important fiber property that influences the wet-laid web strength. The common fiber length measurements are arith-

Table 3. Kajaani FS-300 (Metso) fiber analyzer results

Fiber length	L(n) (mm)	L(l) (mm)	L(w) (Mm)	Coarseness (mg/m)	Fiber curl (%)	Fiber width (micro meter)	Fiber Kink (L/m)	Fines (n)	Fines (L)
Untreated	0.93	2.18	2.93	0.320	17.8	26.22	422.17	38.82%	4.25%
15% NaOH treated	0.98	1.70	2.24	0.366	29.4	25.80	1444.46	18.15%	1.7%

metic mean, a length weighted mean and weight weighted mean[34] as defined by eqs. (6), (7) and (8) respectively.

$$L_n = \Sigma n_i l_i / \Sigma n_i \quad (6)$$

$$L_1 = \Sigma n_i l_i^2 / \Sigma n_i l_i \quad (7)$$

$$L_w = \Sigma w_i l_i / \Sigma w_i \quad (8)$$

where, L_n = numerical average length, L_1 = length-weighed average length, L_w = weight-weighed average length, n_i = number of fibers in *i*-th class, l_i = mean length of the *i*-th class and w_i = weight (or mass) of fibers in the *i*-th class.

Result of untreated sample has a length weighted fiber length of 2.93 mm which was reduced to 2.24 mm after 15% (w/v) NaOH treatment as shown in Table 3. Fiber length reported commercially is using the length weighted mean, and the measured length was between 2 mm to 3 mm which was suitable for preparing absorbent core. Fig. 7 and Fig. 8 shows the fiber length distribution and length fractions for untreated and 15% (w/v) NaOH treated sample. Hence, the procured pulp after NaOH treatment was suitable for preparing the absorbent core.

Fiber width

The dimensions of the pulp fibers depend on the variations in raw material and the pulping processes used. Fibers with larger fiber diameter and thicker walls are stiffer than

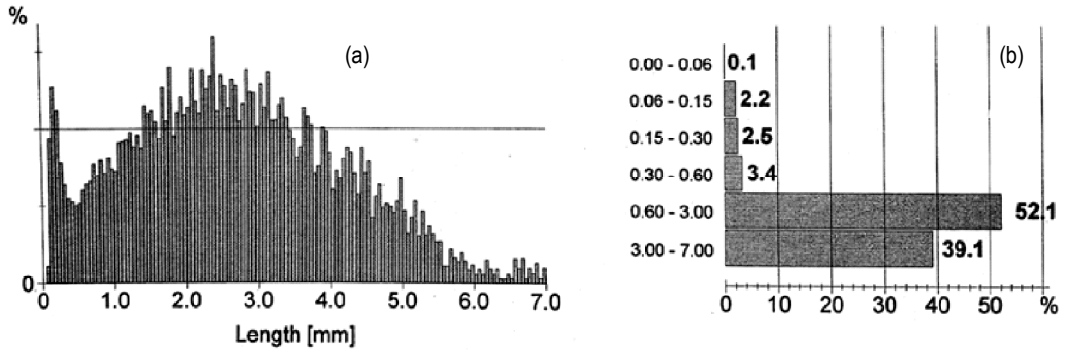


Fig. 7. Length-weighted distribution (a) and length-weighted fractions (b) of untreated sample.

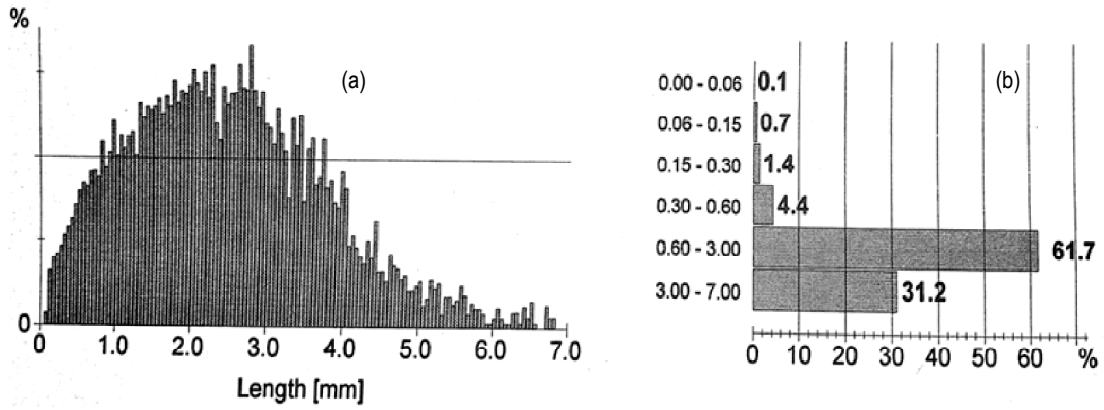


Fig. 8. Length-weighted distribution (a) and length-weighted fractions (b) of 15% NaOH treated sample.

fibers with small diameter and thin walls. Larger and thicker fibers tend to resist compression both in the dry and wet states, providing both resilience and better holding capacity[20]. Typically the length of a softwood fiber is 100 times its width. Fiber width is approximately ten times the wall thickness. When the fiber length of softwood fibers is 3–4 mm, the width is 30–40 μm. The fiber wall thickness is 3–4 μm. The fiber width of untreated and treated pulp is 26.22 μm and 25.80 μm and the width get reduced by 1.60% because the hemicellulose, extractive content and other .impurities get removed from the fiber wall as shown in Table 3. Fig. 9 and Fig. 10 shows the width distribution and fractions of untreated and 15% NaOH treated sample.

Coarseness:

Coarseness is the very important fiber characteristic related to tear strength. Fiber diameter and fiber wall thickness are important as these factors govern the resilience behavior of the fiber, especially in the wet state. Fiber coarseness is the term used for describing these properties. Fiber coarseness is defined as the mass of the oven-dried weight of pulp by the total measured length of the fibers[35]. Fiber coarseness increases after NaOH treatment from 0.320 mg/m to 0.366 mg/m, as shown in Table 3.

Fines:

Fines are the fibers that are less than 0.20 mm in length which is expressed as a total percentage of fiber based on arithmetic basis or length weighted basis. Fines and their amounts can be large depending on fiber morphology, pulping conditions and the extent of mechanical treatment. Fine contents expressed as percentages of the population distributions are not meaningful either, because pulp fibers are used mostly by weight and seldom by number. Literature shows the hand sheets of pulp produced with the addition of an increased percentage of fines and the result demonstrates the addition of 8.2% of fines reduces its air-permeability to one-thirtieth. Fines cause a drastic reduction in sheet porosity and freeness which results in poor dewatering during sheet formation and results in prolonged drying conditions[37]. Fines content should not be very high as this could cause dusting

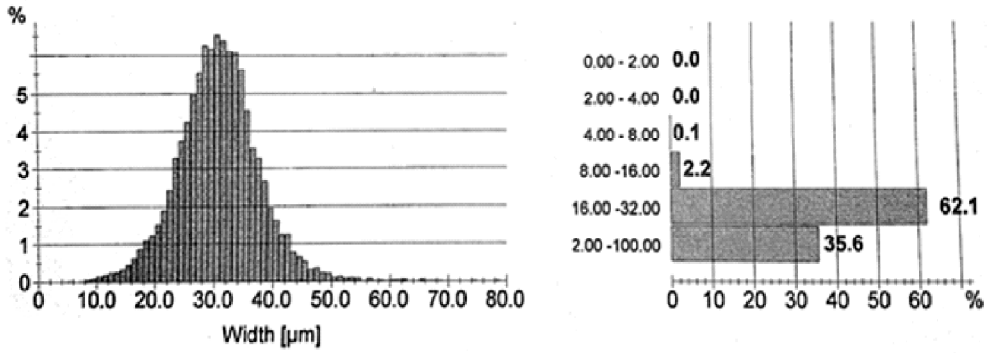


Fig. 9. Width-length weighted distribution (a) and length-weighted fractions (b) of untreated sample.

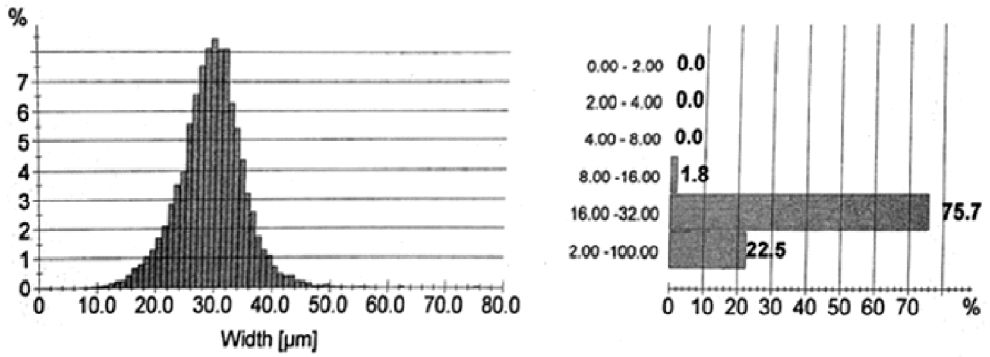


Fig. 10. Width-length weighted distribution (a) and length-weighted fractions (b) of 15% NaOH treated sample.

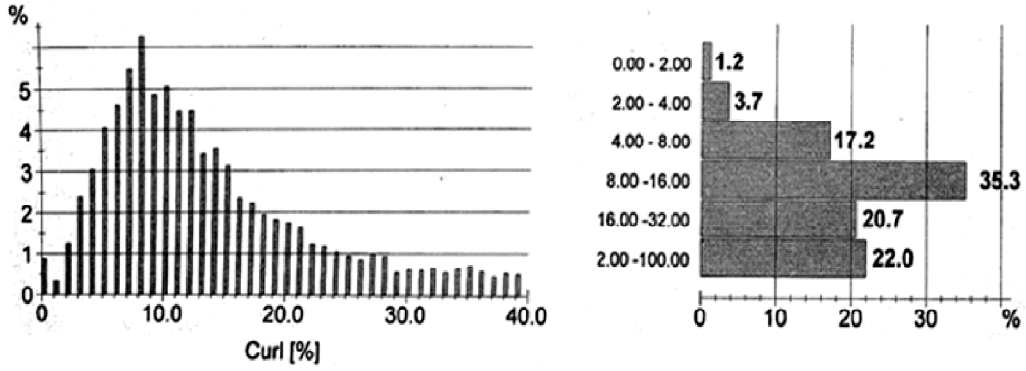


Fig. 11. Length-weighted curl distribution (a) and length-weighted fractions (b) of untreated sample.

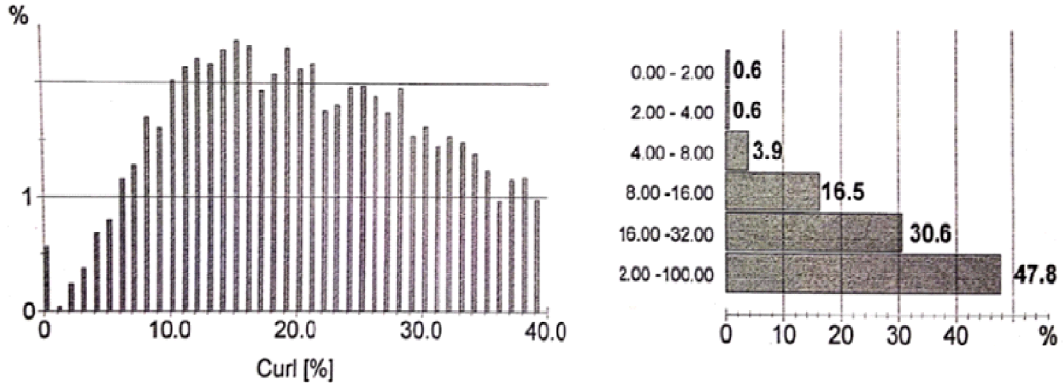


Fig. 12. Length-weighted curl distribution (a) and length-weighted fractions (b) of 15% NaOH treated sample.

problems in the process and decrease pulp quality. The fines content gets reduced after NaOH treatment. The result and Figs. 7 and 8 for fiber length fraction also indicates a reduction in fines(l) from 4.25% to 1.7% as shown in Table 3, this reduction in fines gives improvement in the web quality.

Curl and kink:

Alkali treatment deforms the fiber and induces curl and kink that influence the fiber length. The difference between curl and kink is that fiber in a curl bends at a gentle curve in a kink that changes direction sharply and forms an angle in a fully automated measurement system. Fiber curl is computed for each measured fiber by dividing the Euclidean distance between the endpoints by the length of the fiber along its medial axis. Findings in literature show, an increase in the curl of the fibers resulting in an increase in the critical strain where the critical strain is the strain at which the network ruptures. When the network starts deformation, curled fibers begin to straighten before any slippage between them takes place. There is a clear tendency for alkali treatment to induce curl formation. An increase in the curl index when fibers are treated with an 8% NaOH solution has been already noted[11,35]. Fig. 11 and Fig. 12 shows the length weighted and length weighed fraction of curl distribution indicating increase in the curl % increases for the NaOH treated pulp from 17.8 to 29.4% (Table 3) and the Kink increases from 422.17 to 1444.46 L/m (Table 3).

Conclusion

The study carried out is a novel approach since only a single species of pulp fiber is used for the preparation of absorbent core which would ultimately enhance the performance of the product. The results of the total absorption capacity of the 15% (w/v) NaOH treated wood pulp show improved absorbency by the removal of extractive content. The softness of the pulp is improved by the removal of hemicelluloses and lignin content in a 15% (w/v) NaOH treated sample. This would provide increased absorbency and enhanced comfort to the absorbent core. The results of changes in the

fiber morphology for the 15% (w/v) NaOH treated sample influences on the integrity, strength, wet resilience and flexibility of the absorbent core. The investigation results show the suitability of alkali treated fully bleached softwood (pine) kraft pulp for preparing the absorbent core by using the wet-laid nonwoven technique. In the future study successful preparation of wet-laid absorbent core has been carried out and the absorbency behavior results proved its novelty.

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