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Fiber extraction from *Calotropis gigantea* stem with different retting methods and its comparision

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Abstract
Since ages plants and animal products have been our major source of clothing. The use of natural fibers provides several advantages including low density, low cost, good specific mechanical properties, bio degradability. Currently, one of the major challenges in textile industries is the related environmental problem. The industry is facing great pressure to reduce pollutant emissions. This drives textile manufacturers to seek new approaches to produce environment friendly products. More and more attention has been drawn to agricultural products, wastes and derivatives, because of their renewability. Calotropis gigantea is a common weed in open waste lands, roadsides and railway lines, as well as village surroundings. It grows especially on littoral sandy soils and dry uncultivated land, with periodic dry periods. Considering this prospect a study was carried out to optimise the fiber extraction process with different retting methods like water retting, chemical retting (urea, sodium hydroxide) and enzyme extraction process. Among which chemical retting with sodium hydroxide method showed highest fibre yeld with 5.2%. It was found that Calotropis fiber was completely soluble in strong but partially soluble in NaOH and Na2CO3 solution and Calotropis fibers could withstand the solvents at room temperature. The fiber obtained by water retting method had longer fiber length though it decreased during scouring and bleaching process. Thickness, fineness and bundle strength of fiber extracted with enzyme treatment method was higher than the other methods. Raw fiber extracted by chemical retting with urea has the highest elongation though it has been decreased after scouring and bleaching. Moisture regains of Calotropis fiber after scouring and bleaching was higher than raw fiber and cotton fiber.

Keywords: calotropis gigantea, retting, fiber extraction, biodegradable

1. Introduction
Natural cellulose fibres have successfully proven their qualities when also taking into account an ecological view of fibre materials. Different cellulose fibres can be used for textile and technical applications, e.g. bast or stem fibres, which form fibrous bundles in the inner bark (phloem or bast) of stems of dicotyledonous plants, leaf fibres which run lengthwise through the leaves of monocotyledonous plants and fibres of seeds and fruits.
Calotropis gigantea R.Br. Asclepiadaceae, commonly known as milkweed or swallow-wort, is a common wasteland weed. *Calotropis* belongs to *Asclepiadaceae* or Milkweed or Ak family. *Calotropis gigantea* is a common weed in open waste ground, roadsides and railway lines, as well as village surroundings. It grows especially on littoral sandy soils and dry uncultivated land, with periodic dry periods.

Extraction of fibres from the plant stems is achieved by various methods. Retting is the process which helps in removing of fiber from the stem with the help of chemicals or microbes present in retting water. Retting process involves controlled degradation of the plant stem to allow the fibre to be separated from the woody core and thereby improving the ease of extraction of the fibres from the plant stems. This study was selected to know about the different retting methods for the extraction of *Calotropis gigantean bast* fiber.

2. Materials and Methods

2.1 Selection of plant source

Selected C. *gigantea* that is widely growing plant in wasteland; it grows up to 4 m tall and has clusters of waxy flowers that are either white or lavender in colour. Peeled bark of tender stems of C. *gigantea* plants were selected for the study.

2.2 Fiber extraction process

Retting is a well studies method of extraction of fibers by a natural microbial process. Retting involves the degradation of non-fibrous matter which acts as glue between the fibers in woody plant parts and fibers without damaging the fiber cellulose. This process allows easy separation of individual fiber strands and the woody core.

Since retting is a biological process, it requires both moisture and a warm temperature for microbial action to occur (Hulle *et al*., 2015) [3]. In the present study 4 different retting methods water retting, chemical retting with urea and Sodium hydroxide and enzyme retting are used to extract fiber from the C. *giantea* bark.

2.2.1 Water Retting

Water retting is a wet process by which the bundles of cells in the outer layers of the stalk are separated from non fibrous matter by the removal of pectin and other gummy substances. Water retting may require large amount of clean water and therefore is expensive but results in high quality fibers (Mwaikambo, 2006; Zhang, 2003) [5, 11].

Water retting was carried out in trays filled with clean tap water at room temperature. The Calotropis peeled barks were steeped in water. The weighed Calotropis bark were steeped in water 1 day, 2 days, 3 days, 4 days, 5 days, 6 days, 7 days, 8 days.

2.2.2 Chemical Retting

Chemical retting involves immersion of plants in a tank with a solution of chemical such as sodium hydroxide, sodium carbonate, high pH agent etc. The fibers loosen a few hours but close control is required to prevent deterioration and damage to the fibers.

2.2.2.1 Chemical Retting with urea

Urea is an organic compound that is highly soluble in water and non- toxic also. It is commonly used fertilizer for most of the agricultural crops science it aids the growth of bacteria in soil and water. Urea increases the wetting action of water and enhances the growth of microbes in water (Dhanalaxmi and Vastrad, 2013) [2]. Therefore, urea was used for the extraction of fiber. Urea is added for quick retting.

The bark from retting tray was regularly checked by pressing them within fingertips to see whether the fibers are loosened and can be extract. At the end of retting bark was taken out and the fiber was mechanically extracted by washing in cold water and dried (Banik *et al*., 2011) [1].

The peeled bark were tied in small bundles and immersed in cold water with 0.5%, 1%, 1.5%, 2% urea. The whole process was done at the room temperature. The weighed Calotropis bark was steeped in water up to 6 days.

2.2.2.2 Chemical Retting with alkali (NaOH)

The outer skin of the bark was peeled from the stems by hand and used for fiber extraction. The inner bark was very tough and not suitable for extracting fibers. The peeled bark was dipped in sodium hydroxide solution with a solution to bark ratio of 10:1 at room temperature overnight. The solution was then heated to 80°C for 30 min. The extracted components were then drained and the fibers formed were thoroughly washed first in warm and later in cold water, neutralized in dilute acetic acid solution to remove any remaining alkali, and air dried (Reddy *et al*., 2009) [7].

The bundle of bark were soak overnight in 0.25 N, 0.5 N, 0.75 N, 1.0 N Sodium hydroxide solution (alkaline solution).

2.2.3 Fiber Extraction with enzyme treatment

A method given by Yang and Reddy (2012) [10] was followed for fiber extraction with enzyme treatment.

2.2.3.1 Extraction with alkali solution

Peeled bark of the Calotropis stem was treated with sodium hydroxide at concentration of 1N for 40 minutes at 100°C. Every 100g bark in 1900ml solution. The treated fibers were washed in water to remove the dissolved substances and neutralized with 10% acetic acid solution. Every 100g fiber in 1000 ml solution. Rinse in water and dried at room temperature.

2.2.3.2 Treatment with enzyme solution

Fiber obtained after the alkali treatments were treated with 0.25%, 0.5%, 1.0%, 1.25%, 1.5% enzyme each xylanase and cellulose. The enzyme treatment was carried out at 55°c for 40 minutes with 100g fiber in 1900ml solution at a pH of 6.0. Fiber obtained after the enzyme treatment were thoroughly washed and dried under in room temperature.

3.1 Scouring

Scouring is the process by which natural oil, wax, gum, fat etc as well as impurities are removed completely as possible. Scouring is the pre treatment process of wet processing technology. Scouring is the first stage of pretreatment.

In order to remove the wax, oil, resin and coloring matter from the fiber, first, all fibers were scoured by standard method with a solution of sodium carbonate (4%), sodium hydroxide (1%) and wetting agent (0.5%) at 75°C for 0.5 hour. Percentage was based on the weight of the material, in the liquor ratio of 1:15 (Trotman, 1984) [9].

3.2 Bleaching

Bleaching is chemical treatment employed for the removal of natural colouring matter from the substrate. It is a necessary process to remove the natural and artificial impurities in fibers to obtain clear white fiber. Bleaching with hydrogen peroxide is the most environmentally friendly way to whiten fabrics.

Bleaching of scoured Calotropis fiber was done in a vessel. The samples were boiled with hydrogen peroxide (1%), sodium silicate (1%), soap oil (0.5%) and turkey red oil (0.5%) for two hours. The material to liquor ratio is being maintained at 1:20. After the treatment, the samples were thoroughly rinsed once with hot water and twice with cold water. The samples were then dried in shade (Managooli *et al.*, 2013) [6].

4. Result and Discussion

4.1 Extraction process of *Calotropis gigantean*

Table 1: Yield of fiber from different fiber extraction methods

Water retting		Chemical retting with urea		Chemical retting with sodium hydroxide		Fiber extraction with enzyme treatment	
Days of retting	Weight of fiber (%)	Concentration (%)	Weight of fiber (%)	Concentration (N)	Weight of fiber (%)	Concentration (%)	Weight of fiber (%)
2	0.818	0.5	2.459	0.25	4.682	0.25	4
3	0.989	1	2.545	0.5	4.794	0.5	4.304
4	1.632	1.5	1.955	0.75	5.152	1	4.343
5	3.264	2	1.762	1	5.281	1.25	4.302
6	3.786	-	-	-	-	1.5	4.303
7	2.009	-	-	-	-	-	-
8	1.711	-	-	-	-	-	-

Table 1 represent the yield of fiber on water, chemical and enzyme extraction process. In water retting 6 day soaking gave 3.8% fiber which is the highest. While after 6th day amount of fiber yield is decreased. In chemical retting the amount of fiber obtained is highest (2.5%) in 1% urea treatment. Fiber extraction with enzyme (1%) treatment method gave 4.3% fiber which is the highest yield.
In chemical retting with sodium hydroxide method, the amount of fiber obtained is highest in 1 normality sodium hydroxide which is 5.2%. The chemical retting with sodium hydroxide method showed highest yield of fiber compared to other 3 methods of fiber extraction.

4.2 Morphological features of Calotropis bast fiber

4.2.1 Visual examination of Calotropis bast fiber

This refers to the look and feel of a fiber as observed by the eye and hand. The *Calotropis gigantea* bast fiber was cream in color and twiny. After scouring and bleaching the fibers developed a lustrous white color with a silky soft texture.

Fiber before scouring and bleaching

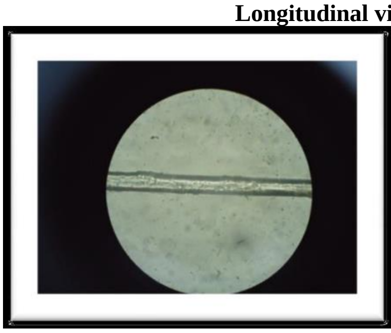
Fiber after scouring and bleaching

Plate 1: *Calotropis gigantea* stem fiber

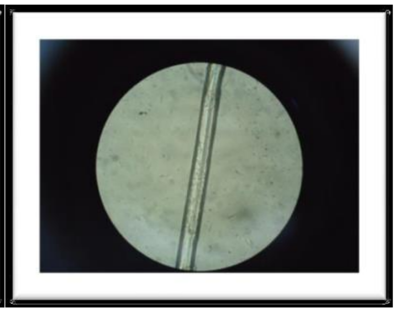
4.2.2 Microscopic appearance - Longitudinal view

Plate (2) shows the longitudinal features of untreated Calotropis bast fibers extracted by different methods. All four slides show a layer of surface deposits which are reported to be composed of lignin, hemicelluloses, wax and other non-cellulosic substances that cover the cellulose inside as stated by Reddy and Yang (2005) [8]. Among the entire four slides second slide shows a clear view which is extracted by chemical retting with urea than the other slides. The longitudinal section of Calotropis bast fiber has a smooth, uniform, high luster and transparent structure.

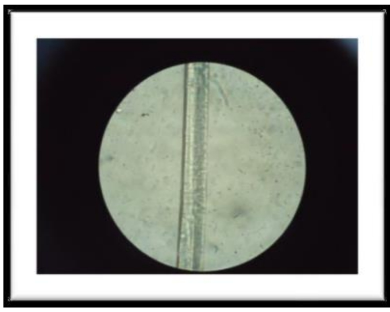
Longitudinal view of raw fiber



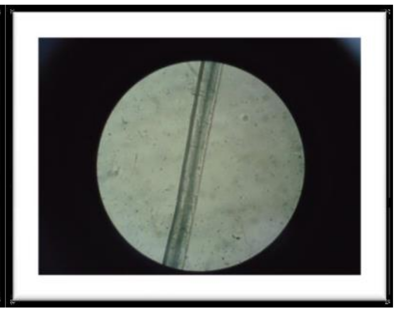
Water retting



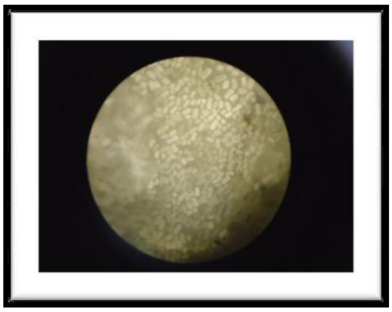
Chemical reting with urea



Chemical retting with NaOH



Fiber extraction with enzyme treatment



Cross sectional view of raw fiber

Plate 2: Microscopic view of *Calotropis gigantea* bark fiber

4.2.3 Burning tests of Calotropis fiber

Calotropis fibres were tested for ignition with flame in which case, the fibres behaved as cellulosic fiber producing odour of burning of vegetable materials like cellulose or paper and also it leaves a gray colour ash as reported by Kalita (2004) [4].

4.3 Chemical properties of Calotropis and cotton fiber

Table 2: Effects of acids on Calotropis and cotton fiber

S. No.	Acid	Concentration	Calotropis fiber		Cotton fiber	
			Room temperature	Boiling temperature	Room temperature	Boiling temperature
1	HCl (Hydrochloric acid)	60%	Insoluble	Partially soluble	Insoluble	Partially soluble
		80%	Insoluble	Soluble	Partially soluble	Soluble
		100%	Partially soluble	Soluble	Soluble	Soluble
2	H2SO4 (Sulphuric acid)	60%	Soluble	Soluble	Insoluble	Partially soluble
		80%	Soluble	Soluble	Soluble	Soluble
		100%	Soluble	Soluble	Soluble	Soluble
3	HNO3 (Nitric acid)	60%	Insoluble	Partially soluble	Insoluble	Partially soluble
		80%	Insoluble	Partially soluble	Partially soluble	Soluble
		100%	Partially soluble	Soluble	Soluble	Soluble

Table 2 indicates that Calotropis fiber was less soluble in strong acids than cotton. Calotropis fiber is partially soluble in 100% of hydrochloric acid, soluble in 60%, 80%, and 100% of sulfuric acid and partially soluble in 100% of nitric acid solution at room temperature. Calotropis fiber is completely soluble in 80%, 100% of hydrochloric acid and 100% of nitric acid. It is partially soluble in 60% hydrochloric acid, nitric acid and 80% of nitric acid at boiling temperature. While cotton was soluble in 100% concentration of all the three acids at both room temperature and boiling temperature and partially soluble in 80% hydrochloric acid, nitric acid at room temperature. Also it is partially soluble in all three acids at boiling temperature.

Table 3: Effects of alkalis

S. No.	Alkali	Concentration	Calotropis fiber		Cotton fiber	
			Room temperature	Boiling temperature	Room temperature	Boiling temperature
1	NaOH (Sodium hydroxide)	12%	Insoluble	Insoluble	Insoluble	Insoluble
		18%	Insoluble	Partially soluble	Insoluble	Partially soluble
		25%	Partially soluble	Partially soluble	Partially soluble	Partially soluble
2	Na2CO (Sodium carbonate)	12%	Insoluble	Insoluble	Insoluble	Insoluble
		18%	Insoluble	Insoluble	Insoluble	Insoluble
		25%	Partially soluble	Partially soluble	Partially soluble	Partially soluble

Table 3 indicates that Calotropis fibers were not affected by 12% and 18% of alkalis but partially soluble in 25% sodium hydroxide and sodium carbonate solution at room temperature. Calotropis fibers were partially soluble in 18% of sodium hydroxide and 25% of both alkalis at boiling temperature. Cotton fiber was partially soluble in 25% sodium hydroxide at room temperature and partially soluble in 18%, 25% sodium hydroxide solution and completely soluble in 25% sodium carbonate solution at boiling temperature.

Table 4: Solubility in solvent

S. No.	Solvent	Concentration (%)	Calotropis fiber	Cotton fiber
1	Acetone	100	Insoluble	Insoluble
2	Carbon tetra chloride	100	Insoluble	Partially soluble

Table 4 shows that Calotropis fibers could withstand 100% solvents. Cotton fibers were not affected by the 100% acetone but partially dissolved in 100% carbon tetra chloride solution.

4.4 Physical properties of *Calotropis gigantea* fiber

Table 5: Effect of different fiber extraction methods and wet processing treatment on mean fiber length (cm)

S. No.	Method	Raw fiber length(cm)	Length of fiber after scouring & bleaching(cm)
1	Water retting	10.94	10.49
2	Chemical retting with urea	9.12	8.33
3	Chemical retting with sodium hydroxide	8.81	8.23
4	Fiber extraction with enzyme treatment	8.68	8.24
Statistical value			
1	SEd	0.120	0.207
2	CD(0.05)	0.586	0.419
3	CD(0.01)	0.786**	0.563**
4	CV %	6.890	5.250

Table 5 shows that calotropis fiber produced from water retting method had longer length (10.94cm) than scoured and bleached fiber. A statistically significant reduction in fiber length was observed in all the four methods of fiber extraction.

Table 6: Thickness of fiber extracted by different retting methods

S. No.	Method	Raw fiber thickness (μm)	Thickness of fiber after scouring & bleaching(μm)
1	Water retting	21.65	16.68
2	Chemical retting with urea	17.14	14.91
3	Chemical retting with sodium hydroxide	25.45	20.03
4	Fiber extraction with enzyme treatment	25.66	20.15
Statistical value			
1	SEd	3.477	2.873
2	CD(0.05)	7.052*	5.876
3	CD(0.01)	9.456	7.814**
4	CV %	35.920	32.840

Table 6 shows thickness of fiber extracted by different retting methods. The table shows that the thickness of fiber extracted with enzyme treatment method was higher than the other methods for the fiber before and after scouring and bleaching.

Table 7: Effect of different fiber extraction methods and wet processing treatment on mean fiber fineness (Micronaire)

S. No.	Method	Raw fiber fineness (mic)	Fineness of fiber after scouring & bleaching (mic)
1	Water retting	5.75	5.69
2	Chemical retting with urea	6.12	6.00
3	Chemical retting with sodium hydroxide	6.28	6.00
4	Fiber extraction with enzyme treatment	6.20	6.60
Statistical value			
1	SEd	0.120	0.128
2	CD(0.05)	0.276	0.295
3	CD(0.01)	0.402**	0.429**
4	CV %	2.390	2.600

Table 7 shows that the fineness of the Calotropis fiber extracted by water retting method was 5.67mic and that of scoured and bleached sample were 5.69mic. Fiber produced by chemical retting with urea was 6.12mic followed by scouring and bleaching was 6.00mic. The fineness of Calotropis fiber extracted by chemical retting with sodium hydroxide was significantly finer (6.28mic) than the untreated fiber (6.00mic). However, Calotropis fibers produced by enzyme treatment after scouring and bleaching were significantly finer (6.60mic) than the untreated fiber (6.20mic). In all the four extraction methods water retting showed the least fineness.

Table 8: Bundle strength and elongation of calotropis fiber

S. No.	Method	Bundle strength (Mpa)		Elongation at break (%)	
		Raw fiber	After scouring + bleaching	Raw fiber	After scouring + bleaching
1	Water retting	16.30	20.93	38.65	23.49
2	Chemical retting with 1% urea	34.86	29.43	53.43	24.26
3	Chemical retting with 1N sodium hydroxide	39.17	77.36	38.80	23.93
4	Fiber extraction with 1% enzyme treatment	42.42	82.12	38.67	23.98
Statistical value					
1	SEd	10.479	23.938	5.564	1.144
2	CD(0.05)	21.860	50.747*	11.796*	2.427
3	CD(0.01)	29.820**	69.921	16.253	3.343**
4	CV %	62.400	72.150	20.760	10.030

Table 8 shows that water retted Calotropis bast fiber has bundle strength of 16.30 Mpa which increase after scouring and bleaching (20.93Mpa). Chemical retting with urea has strength of 34.86Mpa which is higher than the fiber strength after scouring and bleaching followed by chemical retting with 1N sodium hydroxide has strength of 77.36Mpa after scouring and bleaching which is higher than the raw fiber. Fiber extracted with enzyme treatment after scouring and bleaching has the highest fiber bundle strength of 82.12Mpa. Raw fiber extracted by chemical retting with urea has the highest elongation (53.43%) among the four methods. After scouring and bleaching the elongation of fiber extracted by all methods was decreased.

Table 9: Moisture regain and moisture content of *Calotropis gigantea* and cotton

S. No	Fiber particulars	Moisture Regain (%)		Moisture content (%)	
		Calotropis fiber	Cotton fiber	Calotropis fiber	Cotton fiber
1	Raw fiber	23.652	29.003	20.15	23.08
2	Fiber after scouring and bleaching	29.621	25.418	23.23	20.05

Table 9 shows moisture regain and moisture content of Calotropis and cotton fibers. Table shows that moisture regains of Calotropis fiber after scouring and bleaching was more than raw fiber also higher than the cotton fiber. Moisture content of Calotropis fiber after scouring and bleaching was higher than the cotton fiber and raw Calotropis fiber.

5. Conclusion

Chemical retting with sodium hydroxide yields the maximum amount of Calotropis fiber compared to other extraction methods. The untreated *Calotropis gigantea* bast fiber was cream in color and twiny. After scouring and bleaching fibers developed a lustrous white color with a silky soft texture. The longitudinal section of Calotropis bast fiber extracted by chemical retting with urea has smoother, uniform, high luster and transparent structure than other methods. Calotropis fibers behaved as cellulosic fiber produced odour of burning of vegetable materials like cellulose, paper also it leaves a gray colour ash when it subjected to fire.

Calotropis fiber was less soluble in strong acids than cotton. It was completely soluble in 100 percent sulphuric acid. Calotropis fiber is completely soluble in 80%, 100% of hydrochloric acid and 100% of nitric acid at boiling temperature. It was not affected by 12% and 18% of alkalis but partially soluble in 25% NaOH and Na2CO3 solution. Calotropis fibers could withstand the solvents at room temperature. Calotropis fibers were partially soluble in 18 percent of sodium hydroxide and 25% of both alkalis at boiling temperature. Calotropis fibers could withstand the solvents.

The fiber obtained by water retting method had longer fiber length though it decreased during scouring and bleaching process. Thickness of fiber extracted with enzyme treatment method was higher than the other methods though it decreased during scouring and bleaching process. The fineness value of Calotropis fibers produced from fiber extraction by enzyme treatment process was highest. Bleaching after scouring treatment made the fibre finer. Fiber extracted with enzyme treatment after scouring and bleaching has the highest fiber bundle strength among the four methods. Raw fiber extracted by chemical retting with urea has the highest elongation though it has been decreased after scouring and bleaching. Moisture regains of Calotropis fiber after scouring and bleaching was higher than raw fiber and cotton fiber.

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