Asian Journal of Chemistry; Vol. 23, No. 11 (2011), 4925-4931

ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in

Effects of Luffa Cylindirica Fibers on Some Mechanical and Printability Properties of Handsheets

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(*Received*: 15 November 2010; *Accepted*: 20 July 2011) AJC-10182

Luffa cylindrical fibers were both treated with distilled water and acetic acid under an ultrasonic energy media, beaten at a laboratory refiner (PFI) and blended with old corrugated pulps to produce a number of packaging grade paper products. Acetic acid treatment in the ultrasonic energy atmosphere resulted in a higher amount of weight lost (7.2 %) giving thinner and flexible fibers compared to that of treated by distilled water. Mechanical and printing properties of handsheets were negatively affected by the presence of luffa fibers. The surface roughness and the air permeability of resultant papers were also greatly increased by luffa fiber addition. It is concluded that luffa fibers make handsheets quite porous and permeable which directly reduced handsheet strength and printing qualities. Luffa fibers can be perfectly used in the production of permeable packaging materials with the addition of some strength additives.

Key Words: Non-woods, Biomaterials, Luffa, Modification, Sonication, Printability.

INTRODUCTION

Paper recycling and the utilization of alternative raw materials are two most important solutions[1,2] for the shortage of raw materials that pulp and paper industry has been facing. Non wood plants grow fast, have lower lignin content and need less chemicals and energy for pulping. Pulping of non-wood plants is known to be, in general, achieved in 1/3 shorter time with less chemical consumptions compared to wood chips. Therefore countries with limited forest resources have been focused on the utilization of non woody plants, including fiber plants (flax, jute and ramie) and agricultural wastes such as wheat/rice straw, cotton stalks and so on. The use of non wood plants in the pulp/paper production offers many economical and social advantages to countries where the abundant non woody lignocellulose materials are available[2-6].

Luffa cylindrical fibers are not well known in pulp and paper industry compared to that of flax, ramie and linter and so on. Luffa cylindrical fibres are thought to have some potential for paper and fibre board industry. It's natural sponge structure would allow easy and fast penetration of any cooking liquors (compared to wood chips) which would greatly speed up the cooking time. Luffa fibers from its fruits, stems or barks are used in the production of some composite materials, filter and absorbent[7-10]. It is a tropical plant available widely in the wet and warm climates of the world. It naturally grows in Chine, Japan and some other countries in Asia as well as in the border of Mediterranean region including Turkey. The plant produces a fruit with a vascular system in the range of 15 to 100 cm depending on the species and growing spot (Fig. 1)[11,12].

The chemical composition of luffa fibers is similar to that of manila, sisal and hemp (Table-1)[13]. The chemical bonding between fibers as well as between fiber surfaces and composite cement must be created and improved to be able to get a strong and durable bio-composite[14-19]. In order to improve the properties of luffa fibers for composite production, some alkalization[20,21], surface modification[22], acetylation and derivatization studies[12,23] were reported in literature. Hassan also analyzed the anionic exchange capacity and quaternized specialties of luffa fibers[10]. Furthermore, Demir *et al.* did some coatings on luffa fibers with different silane agents to improve the properties of luffa fiber/polypropylene composites[22]. Luffa fiber was reported to be well used in dye adsorption[7,8] and waste water treatment[9] due to its higher surface area and adsorption capacities.

In the fiber modification or some other areas of fiber processing, ultrasonic energy is known to be utilized. Ultrasonic energy is a sound wave with a frequency in the range of 20 to 100 kHz that is well above the human audible range of 16 kHz. Ultrasonic energy is commonly used in cleaning,

Fig 1. (a) Luffa fruits fibers (b) grounded fibers (pulp) distinctive with its whitish colour

TABLE-1
CHEMICAL COMPOSITION OF LUFFA CYLINDRICAL FIBERS[13]

Component	Content (%)
α-Cellulose	63.0
Hemicellulose	19.4
Lignin	11.2
Extractives	3.2
Ash	0.4

degassing, crystallization, dispersion and speeding up some chemical reactions[24-28]. The potential use of ultrasonic in leather, chemical and textile processing industries were extensively studied[29-32] where dying, wash fastness, bleaching of cellulosic fibers (textiles) and tanning of leathers with the help of ultrosound energy were investigated in details in an attempt to design economically and environmentally beneficial methods. Recently Pa[33] used ultrasonic forces during electro-finishing to create a new and high-efficiency method for creating super finishing.

The printability as reflected by print sharpness and density is an important property for a paper product. The print density is strongly related to the amount of colour pigments left on a paper surface after a printing operation is completed. The more pigments on a paper surface stay, the higher print density is obtained. Surface roughness, air permeability, filler content, sizing chemicals are some of paper parameters that significantly influence the print density[34-36]. Ink penetration and print density have a close relationship to ink absorption ability of paper[37]. Senden *et al.* reported that an uncoated paper gives a lower print density than a coated one due to its greater ink absorption[38]. Porosity is one of the most important factors which influence the absorption of ink vehicles. Ink vehicles are drawn into the pores and inter-fiber spaces of paper by capillary action[38,39]. Aydemir *et al.*[36], recently reported that fillers reduced air permeability and surface energy hence increased the liquid uptake. Sizing interfered with the fillers, but had negligible effects on the movements of oil-based ink on papers.

In this work, the potential utilization of luffa cylindirica fibers in packaging grade paper products was investigated as no similar work was found in the literature. Some modification on luffa fibers with acetic acid treatments in ultrasonic energy atmosphere were conducted before refining fibers in a PFI mill to produce pulp. Mechanical properties and print densities of handsheets containing luffa fibers were discussed.

EXPERIMENTAL

Fibers: Luffa cylindrical fibers obtained from the Mediterranean region of Turkey were cut in 2 cm in length, washed with distilled water, air dried and kept in a controlled environment at 20 ± 2 ºC and 65 ± 2 % relative humidity for a couple of days. Fibers for control papers were prepared from an old corrugated board. Board was torn in small pieces and soaked in tap water overnight followed by disintegration in a laboratory pulp disintegrator for 3 min.

Fiber pretreatments: Two treatment protocols were practiced on luffa fibers (LF) as shown in Table-2. Luffa fibers were first immersed in distilled water at 30 ºC for 20 min. This is known as a conventional method (conv.) which is a kind of cold water extraction that takes out easily water soluble impurities and ingredients of fibers. Second protocol, ultrasonic energy and acetic acid treatment followed was relatively harsh on fibers in which luffa fibers were immersed in acetic acid at 30 ºC as well as applying an ultrasonic energy at 20 kHz frequency for 20 min. Brandson B2200B E4 model ultrasonic bath were used in the process. The parameters of acetic acid and sonication treatment were determined from the previous work that extensively discussed the subject[30]. Treated fibers were rinsed with distilled water and allowed to air dry in a conditioned room at 20 ± 2 ºC and 65 ± 2 % relative humidity for 3 days.

Analysis of fibers and pulps: Weight lost on fibers as a result of pretreatments was carefully determined using the equation below (eqn. 1);

TABLE-2
TWO PROTOCOLS WERE APPLIED ON LUFFA FIBERS AS PRETREATMENTS

Methods	Solution	Concentration (%)	Temperature (ºC)	Time (min)	Rinsing process
Conventional	Distilled water	100	30	20	Distilled water
Ultrasonic energy and acetic acid	Acetic acid	100	30	20	Distilled water

$$W_{lst}\ (\%) = \left(\frac{(W_{pre} - W_{after}) \times 100}{W_{pre}} \right) \quad (1)$$

where W_{lst} (%) is the percentage of a weight lost that was extracted from fibers during treatments. W_{pre} is the initial weight of oven dried fibers while W_{after} is the final weight of oven dried fibers after the pretreatment.

Fibre freeness (shopper riegler) and optical properties of sheets (by Elrepho) were determined. SEM images and ATR spectra of fibers were also taken by utilizing Jeol JSM-5410 LV, operated at 20 kV and Shimadzu 8300 ATR spectrophotometer respectively.

Handsheet making and testing: Luffa fibres treated by two methods were taken to a mechanical pulping procedure. Fiber bundles in 2 cm length were undergone a further cutting and separation actions in a laboratory mixer at 3 % consistency for 5 min. Fibre suspensions were then refined in a standard PFI laboratory mill for 2 min in accordance with Tappi T 248 sp-08 method (laboratory beating of pulp).

Three pulp suspensions, two of which were from luffa pulps and one from old corrugated board pulp, were prepared at 0.5 % consistency. A number of handsheets from luffa pulps and corrugated board pulp mixtures (Table-3) were produced on a Rapit Köten handsheet machine (RK). Handsheets were made at 100 g/m^2 grammage in accordance to Tappi 205 sp-95 method (handsheet forming for laboratory tests). Diluted fibre suspension at 0.3 % consistency was filtrated on a 200 mesh forming wire of the RK giving a wet fibre mat. Fibre mat was then placed between blotter papers and wet pressed by rolling a 10 kg cyclinder forward (4 times) and backward (3 times). Handsheets were further hot dried at 105 ºC in the drying part of the RK until reaching 5 % moisture level. Papers were kept in a controlled room set at 23 ± 2 ºC and 65 ± 2 % relative humidity for at least 24 h before testing. The tests done to determine some properties of papers were tabulated with relevant standards in Table-4.

TABLE-3
LUFFA PULPS WERE ADDED TO THE CONTROL PULP AT 15 AND 30 % RATIOS

Handsheet name	Corrugated board pulp (%)	Luffa pulp (%)
Control	100	0
Conventional (15 %)	85	15
Conventional (30 %)	70	30
Ultrasonic energy and acetic acid (15 %)	85	15
Ultrasonic energy and acetic acid (30 %)	70	30

TABLE-4
TESTS AND METHODS FOLLOWED DURING PAPERMAKING AND ANALYZING

Tests	Standards
Beating/pulping	Tappi T 248 sp-08 (PFI method)
Pulp freeness (SR°)	Shopper Riegler method
Handsheet making	TAPPI T 205 sp-95
Paper conditioning	TAPPI T 402 om-88
Density (g/cm^3)-bulk ness (cm^3/g)	TAPPI T-220
Optical properties (ASTM-ISO)	TAPPI T 452 om-92
Air permeability (mL/min)	TAPPI T 460
Surface roughness (mL/min)	ISO 8791-2 (Bendtsen)
Tensile index (Nm/g)	TAPPI T 494 om-88
Breaking length (m)	TAPPI T 404 om-87
Burst index (kPam^2/g)	TAPPI T 403 om-91

Printing and characterization: Handsheets were printed with Michael Huber München Resista Cyan series offset ink (DIN ISO 2746-1) at Heidelberg Printmaster GTO 52 offset printing machine. Printing processes were performed on ten scale of ton and tram values using Wedge 1982 measuring scale control according to ISO 12647-2 method. Reflection values of printed images were measured by using a Gretag Macbeth SpectroEye reflectometer operated at D50 illumination and 45/0 black geometry.

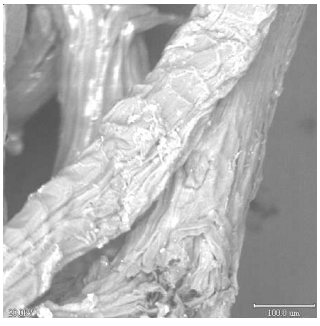
Print density values of tested handsheets were measured with Gretag Macbeth Spectra Eye device adjusted at D50 illumination, 2º monitoring and 0/45 black area[40,41]. Sessile drop method was followed for measuring the changes on drop volume of water on handsheets in a time range[42].

RESULTS AND DISCUSSION

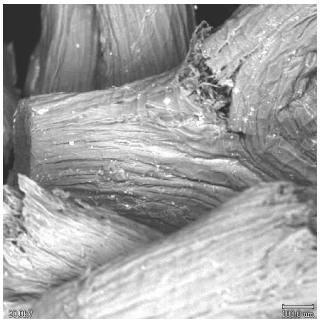
Effects of pretreatments on fibers properties

Weight lost and SEM images: It was seen that the distilled water treatments with and without ultrasonic energy removed 2 and 7 % fibre component respectively. Acetic acid treatments gave 5 % weight reduction while it increased to 20 % with acetic acid and ultrasonic energy method. It shows that treatments removed lignin, pectin and hemicellulose substances from fibres as reported elsewhere[12,21,23,28]. Sonication was believed to increase the solvent (distilled water and acetic acid) penetration into fibres allowing higher extraction and weight lost in fibres.

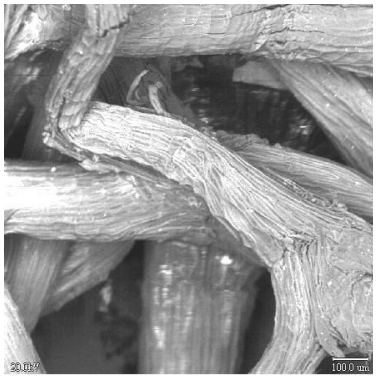
The acid hydrolysis of cell wall component was reported to give a reduction in degree of polymerization, production of sugar molecules[13,17,21]. It is obvious that the application of ultrasonic energy improved the effects of acetic acid treatment as confirmed by SEM images (Fig. 2). Fibres seem to have the destructed cell walls and thinner structures as a result of acetic acid treatment with ultrasonic (Fig. 2).



(a) Untreated



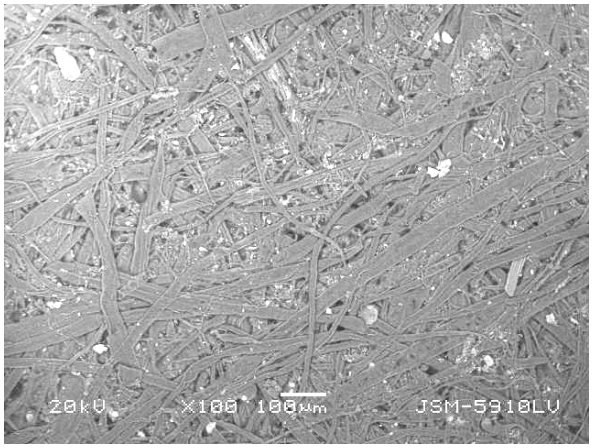
(b) Treated by acetic acid



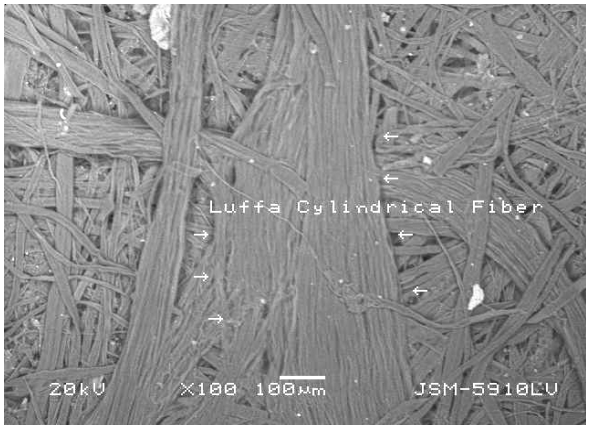
(c) Treated by acetic acid with sonication

Fig. 2. Images of fibres undergone different treatments

Freeness levels of luffa pulps were measured to be as 10, 13 and 17 SRº for untreated, conventional and ultrasonic energy and acetic acid samples, respectively which were in an agreement with SEM images indicating that treatments gave flexible and thinner fibers (Fig. 2). Pulps obtained from old corrugated board cartons had 45 SRº freeness. Pulp freeness is a key parameter which is determined in almost all paper mills to have an idea of drainage during formation and some other properties of resultant paper such as strength and density[43]. It was noted that luffa fibers were quite big in sizes compared to the fibres in controlled pulp and presented as bundles in handsheets (Fig. 3).



(a)



(b)

Fig. 3. Surface images of control paper (a) and paper having luffa fibres (b)

ATR analysis: Tanobe *et al.* extensively studied the characterization of Brazilian Sponge Guard after some chemical applications and prepared a table showing the relevant bonds positions in an ATR spectra for untreated luffa fibers (Table-5)[13]. ATR spectrums of handsheets made in this work were presented in Fig. 4. The acetylation group reaction with acetic acid was previously reported to be occurring preferentially at the easily accessible hydroxyl groups on cell wall of the fibers[23]. The ultrasonic application is believed to improve the accessibility of cell walls for acetic acid and acted as the reaction promoter like a catalyst. The presence of peaks at 1641 cm^{-1} (Fig. 4) indicated the carbonyl group from acetate confirming the acetylation reaction with acetic acid as reported elsewhere[12,23].

TABLE-5
PEAK POSITIONS OF UNTREATED LUFFA CYLINDRICAL[13]

Wave number (cm^{-1})	Assignment
3370	OH stretching
2870	Saturated C–H stretching
1636	Absorbed H_2O
1425	Lignin and CH_2 sym. Bending pyran ring
1314	O–H in plane bending (cellulose)
1155	Antisym. bridge C–OR–Stretching (cellulose)
1110	Anhydroglucose ring
1030	C–OR stretching (cellulose)
894	Antisym., out of phase ring stretching

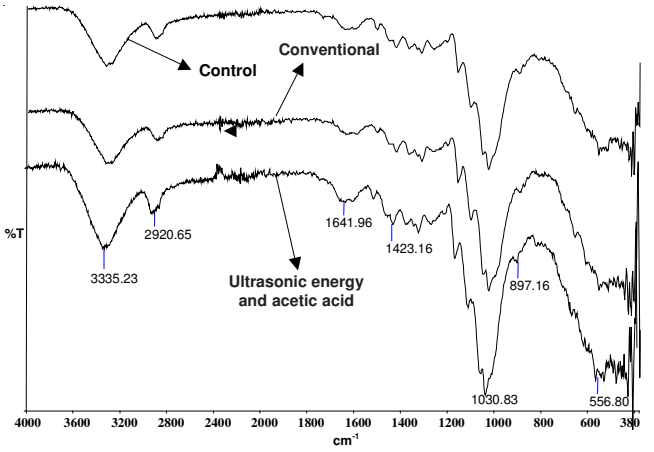


Fig. 4. ATR spectrum of control paper and handsheets containing 30 % luffa pulps treated with conventional and ultrasonic energy and acetic acid methods

Effects of pretreatments on handsheet properties

Optical properties: Luffa pulp has good optical values (Table-6). Acetic acid treatment with ultrasonic energy improved the whiteness from 70.21 to 75.30 and brightness from 60.52 to 65.15. In the paper industry, to be able to get desirable whiteness and bleaching value, the bleaching and deinking methods are applied on virgin pulps and waste papers, respectively. The processes are quite costly and generate some environmental problems, giving stresses on papermills. Luffa pulps in this respect offer a good advantage and it gets even better when implementing simple ultrasonic energy and acetic acid treatments.

Densities, surface roughness and air permeability: Some properties of handsheets made with blend of old

TABLE-6
COLOUR PARAMETERS OF LUFFA PULPS WERE MUCH BETTER THEN THAT OF OLD CORRUGATED PULP

Pulp	Whiteness (ASTM)	Brightness (ISO)	Yellowness (ASTM)	L*	a*	b*
Bleached softwood (reference)	87.51	83.60	5.81	94.95	-0.38	3.21
Luffa pulp (ultrasonic energy and acetic acid)	75.30	65.15	20.87	45.04	1.28	6.21
Luffa pulp (conventional)	70.21	60.52	25.15	48.00	1.43	7.13
Old corrugated pulp	39.37	31.11	30.20	69.02	2.94	11.68

corrugated board pulp and differently treated luffa pulps were presented in Table-7 below. The control paper was made of just pulps obtained from old corrugated boards. No chemical was used in the production. Conventional 15 % refers the papers containing 15 % luffa fibers treated with distilled water as conventional method while ultrasonic energy and acetic acid treatment 15 % is used to indicate sheets containing 15 % luffa fibers treated with acetic acid coupled by ultrasonic energy. It is clearly seen that (Table-7) luffa fibers significantly changed the handsheet properties regardless of treatments they received. The changes become even greater by the increase of luffa fiber content in papers.

Luffa fibers greatly reduced the sheets density from 0.59 to 0.34 g/cm^3 and increased the bulkness from 1.70 to 2.96 cm^3/g (Table-7 and Fig. 5). The greatest change was obtained by the addition of 30 % luffa fibers treated by conventional method. Bundles of luffa fibres seen on paper surface (Fig. 3b) indicate that the treatments did not completely dissolve the middle lamella between fibers. Mechanical pulping with PFI was also not so effective in liberating fibers and creating fibrillations (Fig. 3b) due to most probably the presence of lignin content in Luffa.

The surface roughness is a function of fiber flexibility and fiber coarseness. Paper surface roughness increased from 746 to 2811 mL/min as a result of luffa fibres addition. It means that luffa fibers used here were quite coarse and rigid, hence so difficult to be pressed enough to have a smooth surface compared to pulps of controlled paper.

Air permenance of handsheets significantly increased with the addition of luffa fibres from 850 to 1380 mL/min (Table-7). It suggests that long luffa fibres and fibre bundles (Fig. 3b) could not be pressed well enough to give dense sheet with smooth surface. Therefore, the addition of luffa fibres created some air gaps and galleries in handsheets structure which greatly increased the air permeability of resultant sheets (Table-7).

Strength properties: Luffa fibres addition to the control paper was found to be reducing paper strength properties in significant level (Fig. 6). Luffa fibers were believed to have interfered with the fiber bonding and gave loosely formed fluffy sheets with poor strength properties. I'Anson *et al.* concluded that a paper strength strongly depends on both the

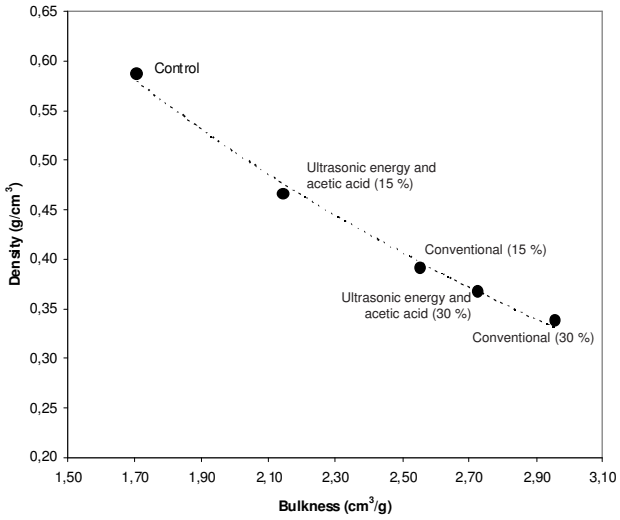


Fig. 5. Luffa pulps greatly reduced the sheets density

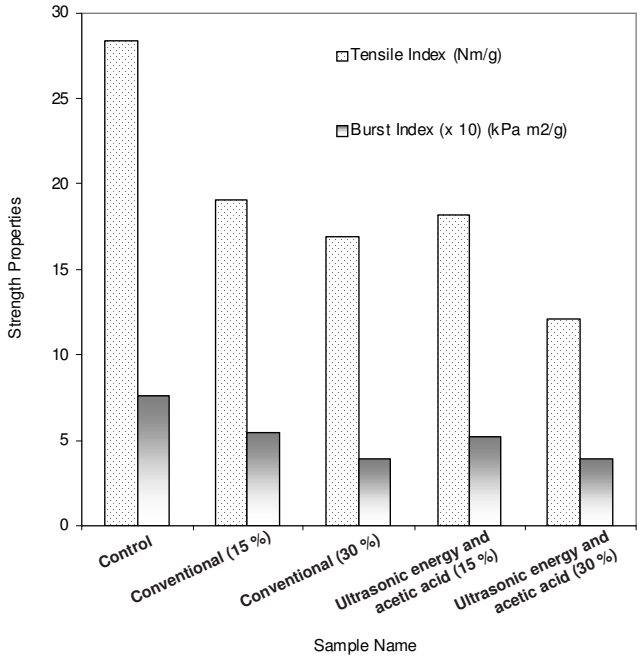


Fig. 6. Luffa fibres reduced the sheet strengths

TABLE-7
SOME PROPERTIES OF PRODUCED HANDSHEETS

Handsheet	Density (g/cm^3)	Bulkness (cm^3/g)	Surface roughness (mL/min)	Air permanence (mL/min)	Tensile index (Nm/g)	Breaking length (m)	Burst index (kPa m^2/g)
Control	0.59	1.70	746	720	28.37	2892	1.52
Conventional (15 %)	0.39	2.55	2461	860	19.08	1945	1.09
Conventional (30 %)	0.34	2.96	2717	1200	16.90	1722	0.78
Ultrasonic energy and acetic acid (15 %)	0.47	2.14	2558	1050	18.17	1852	1.04
Ultrasonic energy and acetic acid (30 %)	0.37	2.73	2811	1380	12.12	1235	0.78

strengths of individual constituting fibers and fiber-fiber bond strengths[43]. Luffa fibers treated with ultrasonic energy and acetic acid method seems to be reducing paper strength slightly more than that of conventionally treated fibers. This is assumed to be probably lower hemicellulose content of luffa fibers due to acetic acid degradation. Ultrasonic energy and acetic acid methods actually made fibers more flexible and finer compared to conv method as revealed by Shopper Riegler freeness test. But in terms of hemicellulose content, the crucial cell components for inter-fiber bonding, ultrasonic energy and acetic acid method was actually believed to be detrimental. Lower the hemicellulose on fiber surfaces; the weaker the interfiber bonding develops between fibers[44].

Relative contact area (RCA) between touching adjacent fibers in a paper matrix is an important parameter for paper strength since it determines the strength of fiber bonding. Relative contact area is also gave some idea of paper porosity. The porous papers normally show lower resistance to air and applied force such as tensile and burst if no extra strength enhancer is used[43-45]. There is a good agreement between the breaking length and the air permanence of hand sheets studied in this work (Fig. 7). It is interpreted that luffa fibers gave porous sheets with higher air permeability and poor strength properties presented as tensile strength, burst index and breaking length (Table-7).

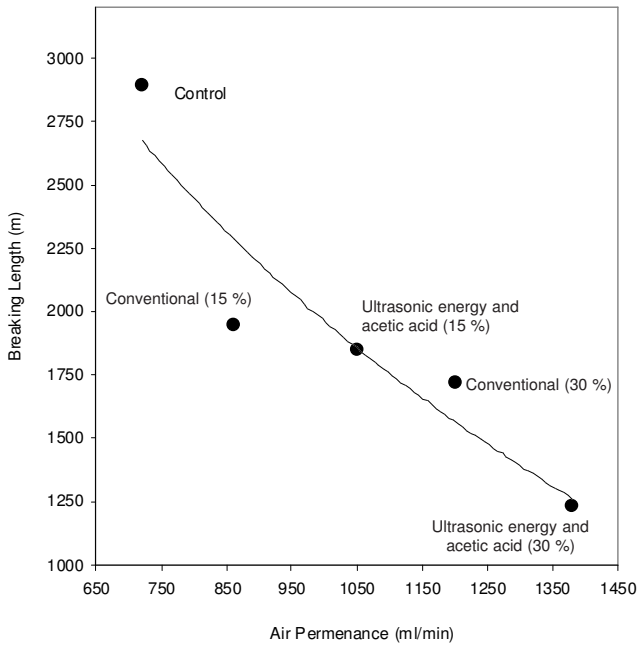


Fig. 7. A good agreement was found between paper porosity and breaking length

Printing properties of hand sheets containing Luffa fibers: Print density is the term used to define the colour intensities of printed images on a material. The ink droplets transferred on a paper get adsorbed into the paper as well as spreading over the surface in time depending on the properties of susbstrate (paper), ink, printing technique and drying methods[37-39]. As seen in Fig. 8, print densities of all papers decreased in different rate and leveled out on 4th day. The greatest changes in print density occurred in a paper containing

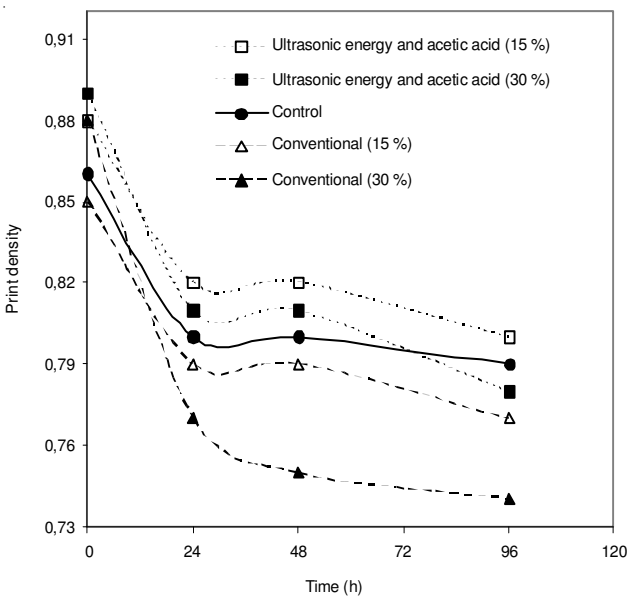


Fig. 8. Conventionally treated luffa fibres gave the lowest print density

30 % luffa fibres treated by conventional method. This was attributed to its highest porosity value as confirmed by air permeabilty test. Sheets with porous structure and absorbent fibres consume more inks and takes more pigment particles which gave lower print density as reported by some researchers[46-48].

The area of contact between an ink droplet and paper surface gets expanded parallel to ink spreading which is defined as tone value. The ink on a paper surface simultaneously travels both in vertical and horizental directions[38,39,48]. Fig. 9 suggests that papers with rough surfaces disturbed inks and gave higher tone values.

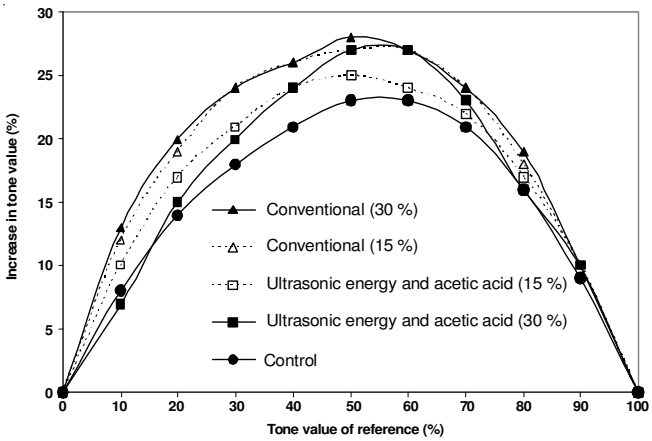


Fig. 9. Luffa fibers increased the changes in tone values

Conclusion

Acetic acid treatments coupled with ultrasonic energy had some positive affects on the quality of luffa fibres to produce papers. In this study, however, the addition of luffa fibres generally reduced the mechanical properties of resultant papers as well as increasing the air permeability and the surface roughness values. Addition of conventionaly treated luffa fibres in handsheets reduced the print densities. Tone values were increased by luffa fibres regardless of treatments they received.

It is suggested that luffa fibers prepared in this study could be better utilized in the production of porous packaging materials and insulation boards with some strength additives hence it greatly increased the bulkiness of materials and reduced the strength. Alternatively, some mild chemical cooking and refining should be followed for producing pulp with better properties for paper making.

ACKNOWLEDGEMENTS

This study was supported by The Marmara University Scientific Research Committee (Project No.: FEN-A-040609-0186, 2009) and (Project No.: FEN-A-040609-0185, 2009)

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