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Assessment of Changes in Corn Husk Fibres after Acid Treatment

Ocena sprememb kislinsko obdelanih vlaken iz koruznega ličja

Original scientific article/*Izvirni znanstveni članek*
Received/*Prispelo* 12-2021 • Accepted/*Sprejeto* 2-2022

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Abstract

Sustainability is desirable in any activity, including farming. Adding value to agricultural wastes such as stover (waste from corn cultivation) would provide financial benefits to farmers while reducing the environmental load of disposal. The literature identifies stover as being a raw material for bio-ethanol and a reinforcement for composites. Fibre from corn husks is generally extracted using an alkali digestion method followed optionally by enzymatic degradation. In this study, acid treatment was investigated for its feasibility to improve the desirable characteristics of alkali extracted corn husk fibres. The results revealed that increasing the acid concentration decreased fibre properties such as average fibre length, linear density and elongation at break. However, breaking tenacity achieved a maximum value, on treatment with 7.5 g/l sulfuric acid, before decreasing. These properties indicate the treatment's adequacy for use in textile products. Acid treatment did not significantly alter thermo-gravimetric analysis values, indicating that the fibre could withstand wet processing conditions.
Keywords: acid treatment, alkali digestion, corn husk, hemicelluloses, lignin, thermogravimetry, TGA

Izvleček

Trajnost je zaželena pri vseh dejavnostih, vključno s kmetijstvom. Dodana vrednost kmetijskih odpadkov, kot so rastlinski ostanki pri pridelavi koruze, bi kmetom zagotovila finančne koristi, hkrati pa zmanjšala obremenitev okolja zaradi odlaganja. Literatura navaja možnosti za uporabo rastlinskih ostankov kot surovino za bioetanol in ojačitev kompozitov. Vlakna iz koruznega ličja po navadi ekstrahirajo z alkalno obdelavo, ki ji lahko sledi še encimska obdelava. V tej študiji je bila raziskana primernost kislinske obdelave za izboljšanje želenih lastnosti alkalno ekstrahiranih vlaken iz koruznega ličja. Pokazalo se je, da so se s povečanjem koncentracije kisline poslabšale lastnosti vlaken, kot so povprečna dolžina, linearna gostota in pretržni raztezek. Najvišja trdnost vlaken je bila dosežena pri koncentraciji 7,5 g/l žveplove VI. kisline in se je z naraščanjem koncentracije začela zniževati. Vlakna so po lastnostih primerna za uporabo v tekstilnih izdelkih. Kislinska obdelava ni bistveno spremenila obstojnosti vlaken pri povišani temperaturi, kar kaže, da so obstojna v razmerah mokrih obdelav.
Ključne besede: kislinska obdelava, alkalna razgradnja, koruzno ličje, hemiceluloze, lignin, termična gravimetrija, TGA

1 Introduction

Nowadays, there is a significant global focus on sustainability, wherein conservation of non-renewable resources is of prime importance. This concept has urged governments, the academia and civil bodies to promote the utilization of renewable natural raw materials. Consequently, such products would be based on sustainable resources, be biodegradable or recyclable and not cause harmful emissions during production, service, or end-of-life disposal [1, 2].
Cotton is the single largest natural cellulosic fibre supplying the global demand of the textile and clothing sectors. The main reason for this is its superior wearer comfort properties. A growing world population needs both clothing and food for its survival. Hence, there is competition for agricultural land between these two basic requirements. This has resulted in intense research being conducted on alternative natural fibres such as flax, sisal, jute, kenaf and hemp [3]. It should be noted that the cultivation of these fibres also needs agricultural land. Hence, lignocellulosic agricultural by-products offer a desirable low-cost alternate source of cellulosic fibres [4].
During the last few decades, lignocellulosic fibres have been considered as substitutes for conventional man-made fibres and for application in fields such as transportation, construction and packaging, in both the academic and commercial arena. These short or long fibres can be used in the form of particulates for fillers, as they possess the properties, composition, structures and features necessary for reinforcements or fillers for polymer composites [5–8]. Utilizing agricultural residues as a fibre source would provide significant benefits for the environment by preventing incineration on the fields. Furthermore, the extra income would benefit the agricultural community [9–11]. The use of farming residues is promising in terms of cost and infrastructure. Production expenditures and land rent are generally covered by the costs of the primary crop. In terms of the infrastructure, farmers or crop processors already have the knowledge, experience, and technology to handle most of the agricultural residues, including corn husks [12–14].
Wheat and barley straw, corn husk, okra, sunflower, corn and nettle stalk and empty banana bunches are examples of agricultural residues that researchers have investigated as sources of fibres [15]. Corn husk has been the subject of limited research in terms of being a fibre source [9, 16]. This is contrary to the abundant availability of corn, the most widely grown grain in the world. Hence, corn husk offers excellent opportunities for research as a source of sustainable fibre [17–19].
The by-products of corn cultivation, namely stalk (50%); leaves (23%), cob (14%) and husk (13%), are commonly termed stover. Stover is usually fed to cattle or more often burnt in the field or used as fuel. Utilization of this by-product would benefit both farmers by providing additional value and the environment by reducing the pollution load of burning. Corn husks, also known as ears or shucks, are fibrous structures that can be up to 20 cm in length and are traditionally used for decoration, food wrapping, and other applications [20–22]. In order to utilize corn husks as a source of fibre, common extraction procedures used for lignocellulosic sources should be considered.
All natural cellulosic fibres other than cotton and kapok are multicellular, with a bundle of individual cells bound by natural polymers such as hemicellulose, cellulose and lignin [17, 20, 23]. The proportion of these components varies depending on age and growing conditions. Fibre characteristics are additionally affected by the extraction parameters employed. Corn husks are composed of about 42% cellulose, 13% lignin, 4% ash and 41% hemicellulose. Similar to bast fibres, the constituents other than ligno-cellulosic fibre can be removed by alkalization [24–26]. Cellulosic fibres are extracted from ligno-cellulosic by-products by using biological, mechanical and chemical methods. The traditional retting process uses bacteria (Bacillus and Clostridium) and fungi (Rhizomucor *pusillus* and Fusarium *lateritium*) found in the environment to remove lignin, pectin and other substances. This process is relatively time consuming and it is difficult to control fibre quality. Alternately, chemical retting uses alkali (sodium hydroxide), mild acids (oxalic acid) and enzymes for fibre extraction. Chemical concentration, temperature and duration of treatment are the main factors determining the quality of extracted fibres [27, 28]. Dilute sulfuric acid is used to hydrolyze hemi-celluloses [29]. Reddy et al. have reported on corn husk fibre extraction by combining alkali digestion and enzyme treatment [30].
The main objective of this study was to observe the effect of acid treatment on alkali extracted corn husk fibre. The extractable fibre content was measured.

Essential physical properties and thermal stability of the fibres were determined according to relevant standards and by using appropriate instruments.

2 Materials and methods

2.1 Materials

Corn husks were collected from farms in the Salem district, Tamilnadu, India. Both outer (matured) and inner (younger) husk leaves were collected after field drying. These husks were grey in color. Sodium hydroxide (100%), acetic acid (98%) and sulfuric acid (98%) were of LR grade and sourced from Sigma Aldrich.

2.2 Methods

2.2.1 Alkaline fibre extraction

10 grams of husk were treated with 10 grams per liter (g/l) of sodium hydroxide at a material to liquor ratio (mlr) of 1:20. The alkali solution was heated to 90 °C before adding the bundle of husks. Treatment was carried out for 30 minutes at boiling temperature. This was followed by rinsing with water at 60 °C and then with water at room temperature. The alkalized samples were neutralized with 2.5 g/l acetic acid at 50 °C and finally washed. The sample was combed using fingers to loosen the fibres and dislodge very short fibres. Subsequent washing removed the impurities. The resultant fibres were dried under ambient conditions and weighed to determine fibre realization. The experiment was conducted in triplicate and the average result is reported.

2.2.2 Acid treatment

Fibres extracted in the previous step were treated, in triplicate, with sulfuric acid to remove residual lignin and hemicellulose. Individual samples weighing 10 grams were treated with sulfuric acid at concentrations of 5, 7.5 and 10 g/l. Consistent treatment parameters of mlr 1:20, temperature 60 °C and time 30 minutes were employed for all three concentrations. The treated fibres were washed twice with water followed by room temperature drying. The weight of fibres before and after treatment was compared to determine the degree of removal of impurities.

2.2.3 Characterization

All samples were conditioned under standard atmosphere of 21 °C and 65% RH for 24 hours prior to characterization. Physical properties such as length, linear density, breaking strength, elongation and moisture content of the extracted fibres were determined in accordance with ASTM standard methods listed in Table 1.

Breaking tenacity was measured using a Tinius Olsen H1KS tester. A crosshead speed of 15 mm/min, gauge length of 2.54 cm. and a 10 N load cell were used. The moisture regain in the fibres was measured using an ETARDY Model 82 & 82/R10 machine. Thermo gravimetric analyses were carried out using a Perkin Elmer TGA analyzer under flowing nitrogen atmosphere (20 ml/min). The samples were scanned from 50 °C to 450 °C at a heating rate of 30 °C/min. The analyses were performed with about 4 mg of air-dried samples.

Table 1: Test methods employed for fibre characterization

Sample no.	Property	Test method
1	Linear density	ASTM D1577-07
2	Breaking tenacity	ASTM D 3822-07
3	Moisture regain	ASTM D 2495-07

3 Results and discussion

3.1 Husk to fibre ratio by alkalization

Alkali treatment yielded 26–28% fibres on the initial weight of corn husks. This is consistent with the work reported by Sari et al [31].

3.2 Weight loss by acid treatment

Table 2 presents the weight loss percentage after treatment at evaluated acid concentrations. The weight loss indicates the removal of impurities such as hemicellulose and lignin and is viewed to be directly related to acid concentration. It may be assumed that the cellulose content was not affected since cellulose is not dissolved by sulfuric acid below 50% (W:W) concentration.

Table 2: Weight loss after acid treatment

Sample no.	H_2SO_4 (g/l)	Weight loss (%)
1	5	9.33
2	7.5	12.53
3	10	13.33

3.3 Fibre length

Length is an important property of textile fibres. In general, a longer fibre is preferred because it confers a number of advantages in the spinning process. Acid treatment reduces fibre length as reported by other researchers [32, 33]. This is commonly attributed to the removal of a large portion of non-cellulosic constituents that glue the fibres together. Average fibre length at different treatment stages shown in Table 3 reveals the inverse relationship between acid concentration and average fibre length. Milder treatment results in longer fibres. Table 4 confirms that corn husk fibre length falls between that of cotton and other vegetable fibres. It should be noted that both alkali and acid-treated fibres have sufficient length to be converted into spun yarns.

Table 3: Fibre length after different treatments

Treatment	Average length (cm)
Alkalized (control)	25.7
H_2SO_4, 5 g/l	24.2
H_2SO_4, 7.5 g/l	19.1
H_2SO_4, 10 g/l	19.9

Table 4: Fibre length comparison

Fibre type	Length (cm)
Cotton	1.6–6.96
Husk fibre	9.5–35.5
Flax	up to 90
Jute	100–400
Hemp	up to 400
Sisal	up to 100

3.4 Linear density

Fineness or linear density is commonly measured in mass per unit length. It influences yarn properties and the resultant fabric properties. Table 5 shows that acid treatment reduced the linear density of corn husk fibres. The degree of reduction is proportional to the concentration of sulfuric acid (H_2SO_4) used. The initial average linear density of 61.7 tex of alkali extracted fibres was reduced to 30.5 tex by acid treatment. A probable reason is the removal of non-cellulosic constituents.

Table 5: Linear density of alkalized and acid treated fibres

Treatment	Linear density (tex)
Alkalized	61.7
H_2SO_4, 5 g/l	43.8
H_2SO_4, 7.5 g/l	38.6
H_2SO_4, 10 g/l	30.5

3.5 Moisture regain

Similar to fineness, moisture regain was reduced after acid treatment in direct proportion to acid concentration. As shown in Table 6, the value is more than that of cotton and less than those of other bast fibres. Table 6 clearly indicates that increasing acid concentration significantly affects the moisture regain values.

Table 6: Comparison of moisture regain of plant fibres

Type of fibre	Moisture regain (%)
Alkalized corn husk	13.5
Acid (H_2SO_4) treated corn husk, 5 g/l	9.7
Acid (H_2SO_4) treated corn husk, 7.5 g/l	10.2
Acid (H_2SO_4) treated corn husk, 10 g/l	11.8
Jute	13.0
Flax	12.0
Cotton	8.5

3.6 Breaking strength and elongation

A fibre must possess sufficient strength to be processed into a textile material. It is widely accepted that a single fibre strength of 5 grams per denier is necessary for a textile fibre although, in exceptional cases, fibre strength around 1.0 gram per denier have been employed [17, 34].

The changes in tenacity and elongation-at-break shown in Table 7 reveal that with an increase in acid concentration, the tensile strength reaches its maximum at the concentration of 7.5 g/l. This may be due to the removal of weak cross-linking material and increased constituent fibre attenuation. The strength became less at higher concentration (10 g/l), probably caused by damage of critical gluing components. Elongation at break reduces at higher acid concentrations.

Table 7: Breaking tenacity and elongation of corn husk fibre

Treatment	Breaking tenacity (cN/tex)	Elongation at break (%)
Alkalized	16.09	7.84
H_2SO_4, 5g/l	16.15	6.91
H_2SO_4, 7.5 g/l	17.98	6.08
H_2SO_4, 10 g/l	13.34	4.16

3.7 Thermal stability

Fibres used in textiles must withstand wet and dry heat, resist ignition on exposure to a flame and preferably self-extinguish when the flame is removed. Heat stability is essential during the manufacturing processes of dyeing and finishing and during cleaning and general maintenance by the consumer. Thermo-gravimetric analysis assesses the change in mass as temperature is increased. Figure 1 indicates that the alkalized and acid treated fibre samples show similar trends of thermal degradation. The initial weight loss observed up to 110 °C is due to moisture evaporation. The degradation of hemicelluloses and other impurities occurred from 250–300 °C whereas cellulose decomposed between 300 and 355 °C. The more thermally stable lignin decomposed at elevated temperatures between 350 °C and 410 °C. Acid treatments did not affect the thermal degradation behavior of the fibres. The observed thermal stability is sufficient for use in textile processes and applications.

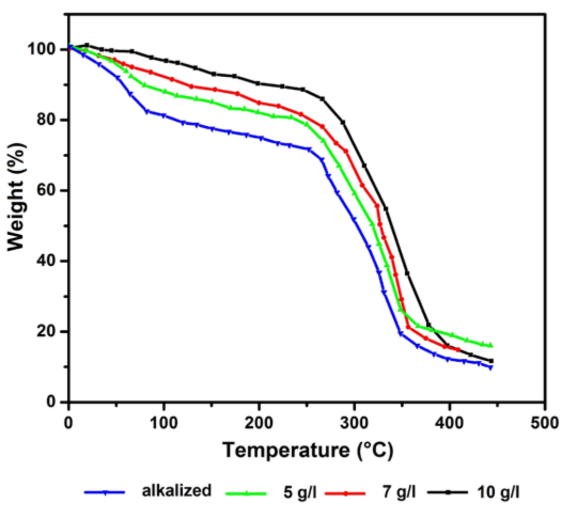

Figure 1: TGA curves of alkalized and acid-treated corn husk fibres

4 Conclusion

Corn husk is a viable source of non-cotton cellulosic fibre. This is of significant importance because husk is a non-avoidable low-value by-product of corn cultivation. The utilization of such a product increases commercial benefits to farmers. In addition, the ecological detriment of burning such waste is avoided. This study is unique in exploring the process sequence of alkaline digestion followed by acid treatment. This approach yielded fibres with characteristics compatible with textile production processes. Work on coloration, blending with other fibres and producing usable commodities is in progress.

Acknowledgements

The authors express their gratitude to the management of their respective institutes for supporting and encouraging this collaborative work.

Conflict of interests

The authors declare that there are no conflicts of interest in the publication of this research.

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