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Original Research Article

Enzymatic Hydrolysis of Rice Straw for Handmade Papermaking

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A B S T R A C T

The principal raw material used for manufacturing paper pulp is wood. However, growing demand in the paper industry, at a time of dwindling forest resources, has compelled the sector to turn to other sources of raw materials, such as cereal straws, reeds, bamboo, or sugar-cane bagasse. Rice straw as alternative raw material in making handmade paper provides an eco-friendly approach in this scenario. Rice straw is available in abundance after harvest. Burning of this agro-residue in agricultural fields causes serious threat to environment. Developing the use of rice straw thus offers many advantages including its commercial use in the mitigation of environmental pollution. However rice straw being a lignocellulosic material is treated with chemicals for pulping that give rise to effluent problem. In the present study, we have developed a method for environmentally safe processing of rice straw by application of certain enzymes.

Keywords

Cleaned, Chopped, Handmade paper, Alkaline earth, Raw material

Introduction

Paper gets its name from papyrus, a reed that the Ancient Egyptians use for making a writing material. They cut papyrus stalk into thin strips and pressed crisscrossed layers of strips into sheets. The paper formed was white, textured and porous. It was claimed that paper was invented in 105 A.D. by Tasi-Lun in Lei- Yung province of China (1,2) Now, researchers have ample evidence to show that the paper was being made in India as back as 250B.C (3). The earliest known account of producing handmade paper as it existed in India is that given by Ironside(4), which gives a complete description of the process and the instruments used. Similar descriptions have been reported in other sources (5). As per the other material reports, the raw material which was usually old ropes, fishing nets, sacking or textiles of hemp, jute or sun hemp, was cleaned, chopped and then pounded by means of a pounder or a levered foot pedal. The pulped material was then washed in the river by suspending it in a cloth attached to the waist of two persons. The

washed pulp was treated with an alkali, usually an alkaline earth. The treated pulp was pounded again. The pounded pulp material was left on platforms of stone or cement to dry and bleach in the sun. This paper was not only used in India but also exported to other countries.

The production of mill made paper began in early 1800s in Europe with the development of paper making machine. The industry is dominated by North American (United States and Canada), Northern European (Finland, Sweden, and North-West Russia) and East Asian countries (such as East Siberian Russia, China, Japan and SouthKorea). Australasia and Brazil also have significant pulp and paper enterprises. However Mill Paper Industry is one of the most polluting industries of the world. Nitrogen Dioxide (NO_2) Sulfur Dioxide (SO_2) and Carbon Dioxide (CO_2) are all emitted during paper manufacturing. Nitrogen dioxide and sulfur dioxide are major contributors of acid rain. Waste water may also be polluted with organo-chlorine compounds. Waste water discharges from a pulp and paper mill contains solids, nutrients and dissolved organic matter such as lignin. It also contains alcohols, and chelating agents and inorganic materials like chlorates and transition metal compounds. Nutrients such as nitrogen and phosphorus can cause or exacerbate eutrophication of fresh water bodies such as lakes and rivers. Worldwide consumption of paper has risen by 400% in the past 40 years with 35% of the harvested trees being used for paper manufacture. In the mill paper, the pulping procedures have to be used with high chemicals and under extreme conditions of temperature and pressure (soda pulping/kraft pulping, sulphite pulping etc. (6)

On the other hand, handmade paper making process is eco-friendly since the pulping procedures utilize minimum chemicals under atmospheric pressure. Handmade paper is often made from tree-free fibers such as cotton, abaca or hemp. Handmade paper is also prepared from recycled fibers viz. mixed office waste, newsprint waste or cotton hosiery waste or textile industry waste (cotton rags). It is labour intensive process. With the continuously increasing demand of Indian handmade paper and its products both in the domestic as well as export market, the number of handmade paper unit has gone up to 450 and accordingly the demand of raw material has gone very high. Getting such a huge quantity of rags may be a difficult task. As a result, availability of traditional raw materials i.e. cotton rags (the traditionally used principal raw material) is getting limited and does not seen sufficient to satisfy the future raw material requirement of the industry (7-8). Under such circumstances, there is a tremendous need of alternative sources of fibers, which can be used as raw material for handmade papermaking. Various agro-residues, bast fibers, stem and leaf fibers hold great promise as a raw material (table-1) supplement of the industry.

Among these large quantities of agricultural residues (rice straw), only a minor portion of the residues is reserved as animal feed. However a huge quantity of the remaining straws is not used and burnt in the fields. The air pollution therefore is a serious problem by burning these residues in this area. Therefore, the use of these straws in pulping or papermaking has many advantages including reducing the need for their disposal and environmental deterioration through pollution and fires (9). From the table-2, it can be seen that the rice straw has very less amount of lignin content so it can be a useful fibrous material for making handmade paper.

Rice is one of the major crops of central and southern China, and 230 million tons of rice straws are generated annually (10)**.** Although

there are a few methods available for rice straw reuse, such as animal feed, fuels for cooking, house heating, biogas etc., a significant amount of rice straw remains unused and burnt in open fields, causing serious environmental and safety problems, such as air pollution and fire disaster. On the other hand, rice straw is an organic material and can be used to produce paper through pulping, thus providing an alternative for rice straw use to mitigate the pollution. Rice straw is easily available in the world so we can use rice straw as the alternative raw material for making paper. India is one of the world's largest producers of white rice, accounting for 20% of all world rice production. Rice is India's prominent crop, and is the staple food of the people of the eastern and southern parts of the country. Rice straw is produced throughout the world as a by-product of rice cultivation. More than two billion people eat rice as a staple food, and the crop dominates cereal production in many low-income countries. The options for disposal of straw are limited and include burning, composting and feeding on-farm. Off-farm, the straw is used for a host of processing activities – livestock feed, compost, pulp, extracts and/or fibers. Straw is also burnt in the home, and sometimes for commercial heat or power. Complex structures make rice straw hard to be biodegraded and used by anaerobic microorganisms and cause low digestion rate and biogas production. Rice Straw is used as biomass. People produced biogas through straw.

As climate change is extensively recognized as a threat to development, there is a growing interest in alternative uses of field-based residues for energy applications. Punjab produces around 23 million tonnes of paddy straw and 17 million tonnes of wheat straw annually. More than 80 % of paddy straw (18.4 million tonnes) and almost 50 % wheat straw (8.5 million tonnes) produced in the state is being burnt in fields. Almost whole of paddy straw, except Basmati rice is burnt in the field to enable early sowing of next crop.

Lately, the farmers have extended this practice to wheat crop also. There is no doubt, the handmade paper production is very eco-friendly but for the sustainable development, natural fibers can be used. Such natural fibers consist the unwanted material called lignin. We have to use certain chemicals for pulping (removal of lignin) of natural fibers. So, as soon as the quantity of these chemicals increases, the effluent problem arises. So, there is an immediate need to develop some ecofriendly pulping and bleaching procedures to minimize the use of chemicals in handmade papermaking. Therefore, keeping in view of the above, present study was taken up to explore the possibilities of enzymatic hydrolysis of rice straw for making handmade paper.

Materials and Methods

Raw material preparation

Chopped rice straw was used as the raw material for present study. It was kept in polythene bags to attain the uniform moisture content and moisture was analyzed before further studies.

Moisture content in the raw material

Moisture of the raw material was determined by weighing 2-3 samples of rice straw in pre-weighed oven dried petri dishes. The samples were then dried in the hot air oven at a temperature of 102 ± 2^{o} C for overnight period or till a constant weight was achieved. After drying, samples were weighed and the moisture content was determined as below:

$$\text{Moisture \%} = \frac{(W_1 - W_2)}{W_1} \times 100$$

W_1 = Weight of sample before drying

W_2 = Weight of sample after drying

Dryness % = 100-Moisture %

Enzyme treatment of rice straw

After moisture determination, samples of rice straw were taken. Out of all samples taken, three samples were treated with different doses of Enzyme &one was the control. The rice straw samples were subjected to enzyme treatment using the conditions as pH 5.5- 6.0, incubation temperature 40℃ and incubation period of an overnight.

After the enzyme treatment, enzyme liquors were collected and stored for analysis. Then the control and treated rice straw was washed with flowing tap water on a muslin cloth.

The washed fibres were squeezed and evaluated for the yield loss, if any. The Rice straw was then subjected to pulping through Open hot digestion, urea pulping and Alkaline Peroxide Pulping (APP).

Cooked Rice Straw pulp was further processed in the following two manners:

Beating Process

Refining Process

Refining or beating of chemical pulps is the mechanical treatment and modification of fibres so that they can be formed into paper or board of the desired properties. It is one of the most important unit operations when preparing papermaking fibres for high-quality papers or paper boards. The beating process was carried out to attain a pulp CSF value of around 350ml. For CSF determination, pulp suspension of 0.3% consistency was used as per the standard TAPPI TEST procedures. The beaten pulp was then used to make handsheets of 60 GSM as per the TAPPI Test procedures for evaluation of the physical strength properties.

Enzyme liquor collection & characterization

The enzyme liquors collected before washing the control and treated fibers and the black liquors collected after the pulping of rice straw straw were measured and subjected to analysis of total solids, pH. Black liquors were also evaluated for Residual Active Alkali (RA).

Total solids in enzyme liquor

For estimating the total solids in enzyme liquor obtained, sample aliquots were weighed in 2-3 pre-dried petri plates and then kept in the hot air oven for 6-8 hrs till a o a constant weight was achieved. The petri plates were cooled in Desiccators before weighing.

$$\text{Total solids (\%)} = \frac{\text{Weight of dried solids}}{\text{Weight of specimen}} \times 100$$

Calculation of fiber and pulp yield

The fiber yield was determined by weighing the total fiber (“W” total net weight of fiber) obtained after treatment. Then moisture content was determined using the two aliquot samples and drying in the oven as briefed earlier.The dryness % was then calculated and OD weight of fiber/pulp was determined as given below:

$$\text{(Oven dry) O.D.} = \frac{W \times \text{Dryness}}{100}$$

The % yield of pulp and /or pulp was calculated as below:

% yield of fiber obtained

$$= \frac{O.D. \times 100}{200}$$

W = Total weight of Rice straw fiber

Pulping of the treated and control fibres

After enzyme treated rice straw was cooked through different pulping methods for removing of lignin from Rice straw. Three types of pulping process were used in this study:

Open digestion

A.P.P (Alkaline peroxide pulping)

Urea pulping

Washing, squeezing and shredding of pulps obtained from open digestion, app and urea pulping

After pulping of the rice straw fiber, the pulping effluent i.e. black liquor was collected and measured and the pulp was then washed thoroughly with tap water followed by squeezing and shredding.

Dryness of pulps

The dryness was determined by weighing the pulp ("W" total net weight of pulp) and taking 2-3 petri plates and weighing it with and without pulp samples.

Then kept it in the hot air oven having temp. 102 ± 2^{o} C for overnight. The dried samples were then weighed and dryness was determined as follows:

$$\text{Dryness in \%} = \frac{(W_1 - W_2) \times 100}{W_1}$$

W_1 = Weight of sample before drying
W_2 = Weight of sample after drying

Yield determination of pulp

The pulp yield was determined by weighing the pulp ("W" total net weight of pulp) and taking 2-3 petri plates and weighing it with and without pulp samples. Then kept it in the hot air oven having temp.102 ± 2^{o} C for overnight. The dried samples were then weighted and pulp yield was determined as follows:

$$O.D. = \frac{W \times Dryness}{100}$$

W = Total weight of Rice straw pulp

$$\text{\% yield of fiber obtained} = \frac{O.D. \times 100}{200}$$

$$\text{\% pulp yield} = \frac{\text{OD weight of pulp obtained x100}}{\text{OD weight of raw material taken}}$$

$$\text{\% of fiber loss} = \frac{(\text{Initial O.D.} - \text{O.D. after Enzyme treatment}) \times 100}{\text{Initial O.D.}}$$

Black liquor characterization

The black liquor obtained from both the pulping procedures was characterized for the parameter including total solids, pH, RAA.

pH of black liquor

It was measured using the pH meter after proper calibration with the standard buffer sol. Of pH-4, pH-7, pH-9.

For this, the liquor was taken out from the cold store and left for some time so as to

attain the room temperature and was then shaken well for mixing. Then the pH electrode was immersed into the liquor to note its pH Valve.

Total solids in black liquor

For estimating the total in black liquor obtained by taking 2-3 petri plates and weighing it with and without pulp samples. Then kept it in the hot air oven having temp. 102 ± 2^{o} C for overnight. The petri plates were cooled in Desiccators before weighing.

$$\text{Total solid (\%)} = \frac{\text{Weight of dried solid}}{\text{Weight of specimen}} \times 100$$

Beating of the pulps

The washed pulp obtained from the digester was subjected to the beating process. The Equipment in used in beating known as Beater**.** The degree of beating was controlled through the CSF measurement. The obtained pulp is beaten to 350 ml. CSF values.

Sheet making

The handsheets of 60 GSM were prepared with the help of British Sheet former as per the Standard TAPPI TEST procedures.

Analysis of Optical and physical Strength Properties of Pulp

Handmade sheets prepared from the control and treated pulps were evaluated for the brightness and physical strength properties as per the standard TAPPI Test procedures.

Results and Discussion

Moisture in the raw material

Moisture in the rice straw was estimated as per the method given before. results are given in the table below it can be seen that the average dryness of the straw was 92.24% which on subtracting from 100came to be a moisture content equivalent to 7.76%.

Enzyme treatment

After moisture analyses, the samples of rice straw designated as control and treated, were subjected to enzyme treatment utilizing the conditions given earlier. After an overnight incubation, enzyme liquors were collected from the respective trays and all the samples were washed properly under tap water.

Fibre yield and total solids in the enzyme liquors obtained after enzyme treatment

Fibre yield was also determined after the enzyme treatment so as to see the extent of fibre loss, if any. The enzyme liquors collected from enzyme treatment were evaluated for total solids (Table-5).

With an aim to make the process of handmade paper making from Rice Straw as raw material, the fiber was subjected to the enzyme treatment using different doses of 0.25%, 0.5% and 1% on oven dry basis with respect to a control set of fiber. The enzyme treated and control fibers thus obtained were further pulped through open digestion, APP & urea pulping using the constant doses of conventional chemicals. The pulps thus obtained were analyzed for physical strength properties and the liquors obtained in different stages were evaluated for the parameters of interest.

Beating of the pulp samples and analysis of the hand sheets produced

All the treated and control fibres were subjected to different pulping procedures of Open hot digestion, Urea pulping and

Alkaline Pulping Process (APP) utilizing the conditions mentioned earlier. All the respective pulps obtained were washed thoroughly, squeezed and shredded to determine the dryness so as to find out the pulp yield obtained respectively. The pulps were then subjected to beating process till a CSF value of 350ml was achieved. The time taken to beat different pulps to this CSF was also noted besides evaluating the strength properties of the hand sheets prepared. All these results are recorded in respective tables as table-6 (for open hot digestion), table-7 (for APP process) and table-8 (for urea pulping).

It can be easily seen from the tables that the enzymatic hydrolysis of the rice straw fibre has proved to be very effective as reflected in the better physical strength properties of the rice straw pulps obtained from open digestion/APP/Urea pulping of the treated straw than the control fibre.

Similarly, the beating energy could also be saved in almost all the treated case as compared to the control case. Such positive effects of enzyme treatment have also been reported earlier but in the case of handmade papermaking from banana fiber(12). The use of rice straw for making handmade paper was also presented earlier (14)**.**

Paper is now a global industry with multinational suppliers managing a complex web of fiber sourcing, pulping and paper production and operation all over the world. To meet growing demand for paper production, the pulp and paper industry is expanding its production capacity, primarily in developing countries with lower raw material and labour costs and looser environment regulations.

The pulp and paper industry has significant impact all around the on people and communities world. It is responsible for violations of land right in many places where the fiber for paper is sourced, both in natural forest and by the establishment of plantations on land without the consent of local people. In, Addition, the demand of paper is expected to increase.

Today the finest of papers are produced all over the world. But one dismaying fact is that millions of trees are fell in a day to make paper. Increased demand of paper production and limited wood resources has directed researchers to look for appropriate additional resources of non-wood materials for pulp and paper manufacturing.

Several kinds of non-wood lignocellulose by products agricultural cultivation have investigated by researchers for paper making. Paper made by conventional methods cause high pollution problem. This disastrous effect will affect over world’s beautiful ecosystem.

The handmade paper industry uses exclusively non-forest raw materials. Mostly, it uses only cellulose-rich materials such as cotton rags, waste paper. This could easily be extended to the use of biomass materials and agricultural residues, some of which can be grown specifically for handmade paper production. Non-wood biomass resources have the additional advantage of being amenable to conversion by environment-friendly processes.

Table.1 Raw materials for handmade papermaking

Raw Materials	Sources	Suitability
Straws(e.g. from wheat, Barley or Rice)	Between 5 and 10% of all straw which is produced is burnt.	Short fibered (1.5mm), it is often mixed with other pulp to provide a suitable pulp stock for a variety of uses
Bagasse	from sugar cane after sugar has been extracted	Slightly longer fiber than straw. Suitable for high quality writing and printing paper.
Maize stalks	Remaining after maize harvest	The high moister content and need for collection make maize stalks suitable only for very small- scale production properties are similar to straw
Cotton	Cutting, lint and fluff from cotton mills	Cotton is a high value fabric and is therefore used only for specialist papers. Has a fiber length of 25-32mm
Rags (cotton, denim waste, hosiery waste)	Collected	Often require sorting and bleaching
Flax	A residue from the manufacture of linen	Long fibres make this material suitable for high quality paper .
Hemp and Sisal	From old ropes and tow from rope making factories	6mm fiber length, processing similar to that of cotton.
Jute	From old sacs	Jute does not bleach well and is therefore used for its strength rather than for high quality grades.

Table.2 Proximate analysis of rice straw (11)

Parameters	Content (%, DM)
Cellulose	33.4
Hemicellulose	28.2
Lignin	7.4
LCH (Lignin, cellulose, and hemicellulose)	69.0
cold-water extractives	15.6
hot-water extractives	17.8
benzene−ethanol extractives	10.4
TS	94.0
VS	87.2
TKN	0.8
TC	41.5
Ash	12.8

Table.3 Conditions Used for Different Pulping Procedures

Parameters	Open Hot Digestion	Alkaline Peroxide Pulping Process	Urea Pulping
Rice straw	150 gm	150 gm	150 gm
NaOH	8%	8%	3%
Urea	-	-	14%
Hydrogen Peroxide	-	2%	-
Temperature	100 ℃	95℃	100 ℃
Time	3 hours	3hours	4 hours
Vessel	Open Utensil	Rotary Digestor	Open Utensil
Bath ratio	1:8	1:8	1:8

Table.4 Moisture in the raw material

Petri Dish	Weight of petridish	Weight of sample taken	Weight of petridish after drying	Dryness in %
A.	36.710	2.991	36.474	92.41
B.	38.504	1.466	39.854	92.08

Avg. dryness = **92.24 %,** Moisture: 100-92.24=7.76 %

Table.5 Evaluation of fibre and liquor after enzyme treatment

Parameters	Control	0.25% enz.	0.5%enz.	1.0%enz.
Fibre yield	98.15%	97.42%	98.05%	94.52%
Total solids in Enzyme liquor	0.62%	0.63%	0.75%	1.92%

Table.6 Evaluation of rice straw pulp,black liquor and beating time from open digestion

Parameters	Control	0.25% Enz	0.50% Enz	1.0% Enz
Yield of pulp (%)	76.2	74.4	72.52	71.35
T.S. of B.L. (%)	1.04	1.79	1.92	2.55
RAA (as NaOH) in gpl	0.352	0.464	0.624	0.72
Beating Time (min.)	7.17	3.09	3.00	1.15
CSF	340	350	340	350
Physical Strength Properties				
Tensile Index (Nm/gm.)	21.14	28.50	20.86	26.79
Tear Index($mN.m^2/gm.$)	5.26	5.84	5.62	5.45
Double Fold (No.)	256	160	146	210
Burst Factor ($Kpa.m^2/gm.$)	15.44	16.83	-	15.42

Table.7 Evaluation of rice straw pulp, black liquor and beating time from APP process

Parameters	Control	0.25% Enz	0.5% Enz.	1% Enz.
Yield of pulp (%)	77.27	78.41	76.86	75.70
T.S. of B.L. (%)	1.55	2.54	2.02	2.86
RAA (as NaOH) in gpl	0.976	0.912	0.892	0.92
Beating Time (min.)	3.44	3.0	3.01	1.40
CSF	340	340	350	360
Physical Strength Properties				
Tensile Index (Nm/gm.)	18.04	19.87	20.52	25.04
Tear Index ($mN.m^2/gm.$)	7.30	8.39	8.57	7.98
Double Fold (No.)	64	65	60	78
Burst Factor ($Kpa.m^2/gm.$)	-	-	-	-

Table.8 Evaluation of rice straw pulp, black liquor and beating time from urea pulping

Parameters	Control	0.25% Enz	0.50% Enz	1.0% Enz
Yield of pulp (%)	80.20	86.53	83.64	85.41
T.S. of B.L. (%)	2.68	1.36	2.48	1.11
RAA (as NaOH) in gpl	0.77	0.67	0.97	0.55
Beating Time (min.)	4.06	5.03	4.85	4.35
CSF (ml)	360	370	350	370
Physical Strength Properties				
Tensile Index (Nm/gm.)	18.05	20.34	26.44	25.6
Tear Index ($mN.m^2/gm.$)	5.58	5.70	7.28	6.4
Double Fold (No.)	222	200	122	150
Burst Factor ($Kpa.m^2/gm.$)	16.36	15.10	17.44	15.5

Fig.1 Control and treated rice straw fibre

Fig.2

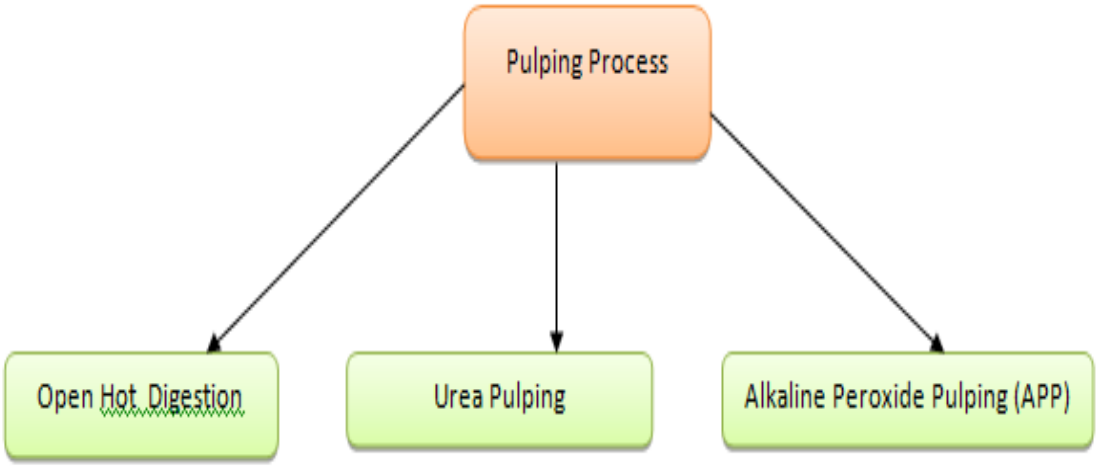

Fig.3 Black Liquor Collected after Pulping of Rice Straw

From the present study, it can be concluded that the enzyme treated fiber of rice straw can be processed in an eco-friendly manner for making tree-free handmade papers thereby reducing the problems of global warming and ozone-layer depletion.

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