

Formic acid pulping process of rice straw for manufacturing of cellulosic fibers with silica

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ABSTRACT: Emerging technology has the potential to develop entirely new approaches for producing cellulose fiber-based materials along with fuels and chemical raw materials like lignin and furfural. Rice straw is a rich source of cellulosic fibers and inorganic micron-sized particles termed as ash. They can prove helpful in development of new or enhanced agricultural residue-based materials and products that offer cost effective substitutes for nonrenewable materials used in different domestic and industrial applications. Lignocellulose is an abundant material that is submicronic at the basic level. Rice straw is a fibrous lignocellulosic material obtained as agricultural residue, but it differs from most crop residues in its high content of silicon dioxide (SiO_2). Ash content on a dry weight basis ranges from 13% to 20%, varying according to the state of conservation of the straw after harvest. The ash in rice straw has nearly 75% SiO_2 . The particle size analysis shows variation from a few microns to hundreds of microns for inorganic residues left after burning at high temperatures above 550°C. Proximate analysis of rice straw shows that it contains 54% to 56% holocellulose and 15% to 18% lignin, both of which are natural biopolymers. The compound analysis shows the different compounds present in rice straw ash.

Rice straw is available in hundreds of million tons in India and other Asian countries, so suitable technologies are required to convert rice straw from a biomass waste to useful bioproducts like pulp, paper, and paperboard. This research paper is intended to obtain pulp with fibers having inherent silica present in it to give high opacity paper and better bonding between fibers.

Application: This research work provides a suitable pulping process for rice straw that can convert high silica in rice straw from a liability to an asset for the pulp and paper industry. The silica remaining in pulp provides high opacity and better bonding in paper.

Lignocellulosic biomass is one of the most abundant biological raw materials and has a nanofibrillar structure. This has the potential to be made multifunctional, and can be controlled in self-assembly. Nanotechnology R&D priorities related to lignocellulosic biopolymers can be categorized as liberation of nano-dimensional cellulose fibrils; directed design of biopolymer composites; nano-sensors; developing processes for synthesizing lignocellulosic-based nanomaterials; and exploiting the nanoscale architecture of lignocellulosics [1]. Cellulose fibers, cell wall fragments, and cellulose microfibrils can be used as reinforcing materials for biocomposite manufacture [2].

Rice straw is a fibrous lignocellulosic material typical of most agricultural residues; however, it differs from most crop residues in its high content of silicon dioxide (SiO_2). Ash content in rice straw, on a dry weight basis, ranges between 13% to 20%, varying according to the state of conservation of the straw after harvest. The ash in rice straw has nearly 75% SiO_2 , 10% potassium oxide (K_2O), 3% phosphorus pentoxide (P_2O_5), 3% iron(III) oxide (Fe_2O_3), 1.3% calcium oxide (CaO), and smaller amounts of magnesium (Mg), sulfur (S), and sodium (Na). The potential

environmental benefits of diverting rice straw from open-field burning to other uses is reduction of air pollutants such as volatile organic compounds (VOC), sulfur dioxide (SO_x), nitrogen oxide (NO_x), and particulate matter (PM10), as well as silica emissions, which are not specifically monitored but can be a health hazard [3,4].

Cooking of rice straw in a mixture of formic acid/acetic acid/water results in the retention of silicon derivatives in the pulp. Study of the various morphological fractions of the pulp by scanning electron microscopy (SEM) equipped with energy dispersive spectrometry shows that the silicon derivatives are essentially localized in epidermis cells, where they occur as silica and calcium silicate [5]. The SEM analysis of dry rice straw has shown that SiO_2 dots are smaller than 1 μm in diameter. Pyrolysis of SiO_2 results in formation of silicon carbide (SiC), which has X-ray diffraction (XRD) analysis d-values of 0.250, 0.216, 0.153, and 0.130 nm [6].

Crystalline cellulose present in lignocellulosic raw materials is a glucose-based polymer that is the most abundant organic polymer on earth. It is more resistant than other constituents to being broken down, but could prove to be a useful nanomaterial. These are made of

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cellulose nanofibrils that are roughly 5–20 nm in diameter and hundreds of nanometers in length. Cellulose nanofibrils, cellulose nanocrystals, cellulose whiskers, and nanocelluloses can be used to produce reinforced composites for manufacturing. The stiffness (145 GPa) and tensile strength (7.5 GPa) of these nanofibrils approach those of the carbon nanotubes that are currently used to reinforce materials and should also, one expects, be much cheaper to produce [7]. Cellulose fibrils may be extracted from mass-produced sulfite and sulfate pulp. The fineness of the fibrils varies according to the isolation process (mechanical or chemimechanical). Only mechanical processing of fibrous raw material produces fibril structures several micrometers long and between 20 and 100 nanometers in diameter. Combined chemical and mechanical processes achieve even finer fibril structures of only a few nanometers in diameter [8,9].

Annual world rice (*Oryza sativa*) production was about 755 million metric tons in 2019. Asian farmers contribute nearly 90.6% of total production. India and China produced 176 and 210 million metric tons, respectively, in 2019. Indonesia, Bangladesh, Vietnam, Thailand, Myanmar, Philippines, Brazil, and Pakistan produce 56.9, 54.5, 43.8, 30.3, 26.9, 18.9, 11.1, and 10.9 million metric tons, respectively, of rice annually [10]. For every ton of grain harvested, nearly 1.40 metric tons of rice straw remains in the field.

Rice straw has a high potential for several uses in different industries as a source of lignocellulosic biomass because of the high production of rice straw per hectare. The proportion of recoverable straw depends on the technique of reaping and harvesting (manual or mechanical) and on the condition of the field (wet or dry). About 2.5–3.0 metric tons/acre of dry rice straw is an average net production. Rice straw was usually disposed of by open-field burning because it is a cheap disposal method [11]. At present, a large fraction of rice straw — nearly three-fourths of the crop residue equal to 70 to 80 million metric tons — is disposed of by burning in India. To facilitate delignification of woody materials, use of organic solvents has been greatly expanded during the past few years [12]. Different constituents of rice straw, including holocellulose, α -cellulose, and lignin (namely, 60.7 wt%, 41.2 wt%, and 21.9 wt%, respectively), are similar to those of some woody raw materials like pine and eucalyptus, as well as various nonwood materials [13]. The rationale for this work, progress in developing viable commercial processes, differentiating types of processes, and deduction of chemical mechanisms have been studied. Philosophically, two major commercial approaches have emerged. One centers on isolation of lignin and chemical byproducts in addition to pulp, while the other focuses on pulp only, concurrent with process simplification. Both will likely have their place in the future. However, the huge amounts of residual plant

biomass considered as “waste” can potentially be converted into various different value-added products like natural fibers used for papermaking, biofuels, and chemicals [14].

FORMIC ACID PULPING OF RICE STRAW

Rice straw pulping with formic acid was studied for different temperatures, cooking times, and acid concentrations. A delignification percentage of approximately 85% with a pulp yield of 44.4% was obtained under relatively mild cooking conditions (temperature, 100°C; cooking time, 60 min; formic acid concentration, 90%). Pulp chemical and mechanical properties were comparable with those found for pulp obtained in basic environments. However, the advantage of this technique compared with cooking in basic environments is that most of the silicon derivatives remain in the pulp [15].

Several variations on acetic acid pulping and optimization of process parameters have been developed over the years, and several new acetic acid-based processes are currently under development [16]. Mineral acid catalysts have been utilized to varying degrees of success. Lignin removal and recovery are analyzed. The importance of swelling the lignocellulosic gel is emphasized for lignin removal. The molecular weight characteristics of lignin from acetic acid-based pulping of wood are described in relation to liquor composition and degree of delignification. It appears that rapid delignification is associated with removal of large lignin fragments [12].

Production of high-purity pulps from *Eucalyptus globulus* or aspen wood via pulping in organic acid media (Milox or Acetosolv technologies) and totally chlorine free (TCF) bleaching have been studied [17]. Solvent pulping methods can be divided into “uncatalyzed,” acid-catalyzed, and base-catalyzed processes. The rates of acid-catalyzed processes appear to be governed by the hydrolysis of α -ether bonds in lignin [18]. Wood processing in hydrochloric acid (HCl)-catalyzed acetic acid media (Acetosolv pulping) provides an efficient way to separate the main fractions of the raw material. In a single step, delignification and hemicellulose degradation are reached with good selectivity. The economic feasibility of this approach depends on the recovery of two marketable pulping byproducts: lignin and furfural [19].

Organosolv pulping processes (based on the utilization of organic solvents as delignification media) can be considered as “biomass refining” technologies, since they lead to a solid phase enriched in cellulose and to a liquid phase containing hemicelluloses and lignin-degradation products free from sulfur.

Cellulosic fiber and silica-based composite materials can be produced by using two solid waste materials: rice straw, a cellulosic agricultural residue; and pulverized coal fly ash from thermal power plants, which contains up to 70% SiO₂. These materials can be used in the manufacture of base paper for decorative laminate and other industrial products [20].

Serial No.	Analysis	Average $\pm$ Standard Deviation	TAPPI Standard Test Method
1	1% NaOH solubility, %	35.75 $\pm$ 2.81	T 212 "One percent sodium hydroxide solubility of wood and pulp"
2	Ash, %	17.48 $\pm$ 1.30	T 211 "Ash in wood, pulp, paper and paperboard"
3	Silica, %	13.33 $\pm$ 1.06	T 211 "Ash in wood, pulp, paper and paperboard"
4	Alcohol benzene solubility, %	10.75 $\pm$ 1.42	T 204 "Solvent extractives of wood and pulp"
5	Klason lignin, %	17.34 $\pm$ 0.69	T 222 "Acid-insoluble lignin in wood and pulp"
6	Holocellulose, %	55.35 $\pm$ 0.68	T 203 "Alpha-, beta- and gamma cellulose"
7	Hot water solubility, %	13.49 $\pm$ 0.43	T 207 "Water solubility of wood and pulp"
8	Cold water solubility, %	7.25 $\pm$ 0.45	T 207 "Water solubility of wood and pulp"
9	Moisture content, %	12.80 $\pm$ 0.98	T 258 "Basic density and moisture content of pulpwood"
10	Bulk density, Kg/m ³	113.5 $\pm$ 2.80	UM 23 "Bulk density of woodchips"

I. Proximate analysis of rice straw

Serial No.	Variable	Values
1	Formic acid concentration, %	55, 65, 75, and 85
2	Catalyst (sulfuric acid; H ₂ SO ₄) concentration, %	0.5, 1.0, 1.5, and 2
3	Time of reaction, min	120, 150, 180, 210
4	Liquor-to-straw ratio	8, 10, 12, and 14
5	Temperature, °C	45, 60, 75, and 90

II. Process variables in pulping.

EXPERIMENTAL

Material

Rice straw was procured from agricultural fields in Sangrur (Punjab), India. This straw was washed with water, air dried, cut into 1.0 cm–1.2 cm long pieces, and packed in airtight plastic containers for subsequent analysis and processing. The proximate analysis of the rice straw was carried out using appropriate TAPPI Standard Test Methods. The analysis is shown in **Table I**.

Delignification of rice straw with formic acid

Delignification (pulping) of rice straw was carried out with formic acid as the main chemical and sulfuric acid (H₂SO₄) as a catalyst. The pulping experiments were conducted at various conditions as given in **Table II**.

For each experiment, the pulping process was carried out by putting 100 g of oven-dry rice straw in a steel cylinder and placing it at controlled temperature inside the laboratory research digester. After completion of the specified time, the residual mass was cooled to 55°C–60°C and filtered out.

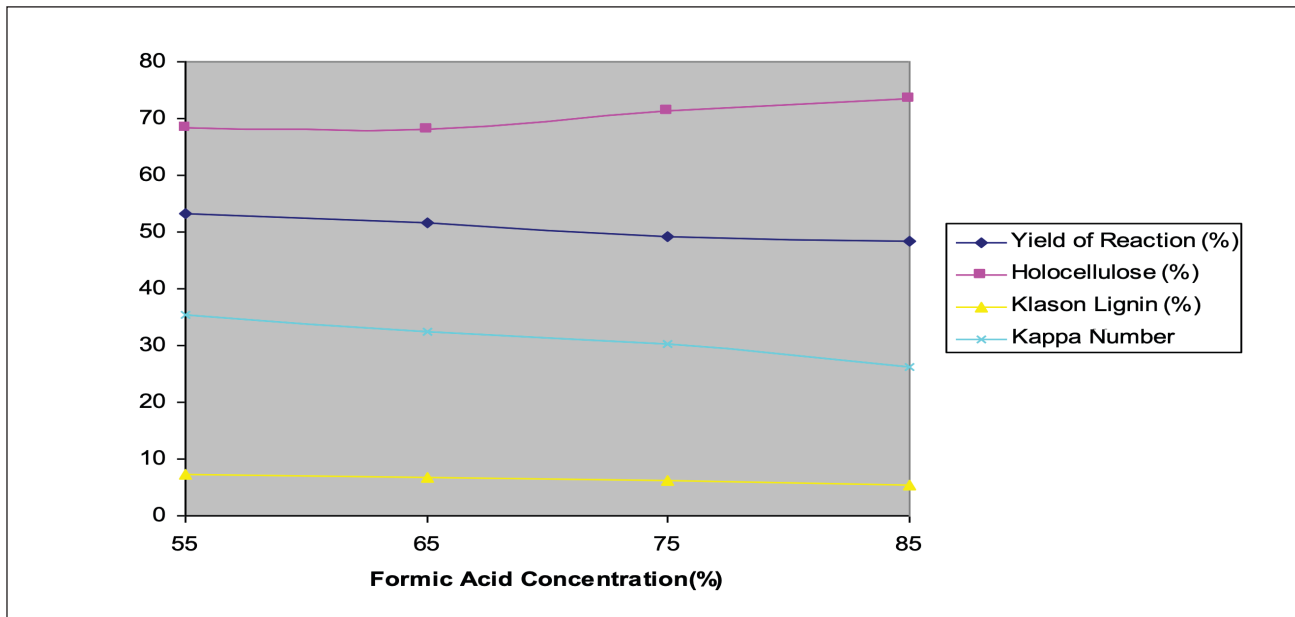
Analysis of the pulp

The pulps obtained after formic acid delignification were analyzed for percentage yield, kappa number, holocellulose, Klason lignin, ash, and silica content. The fibers of pulps were classified by obtaining details of fiber length in a Bauer Macnett fiber classifier.

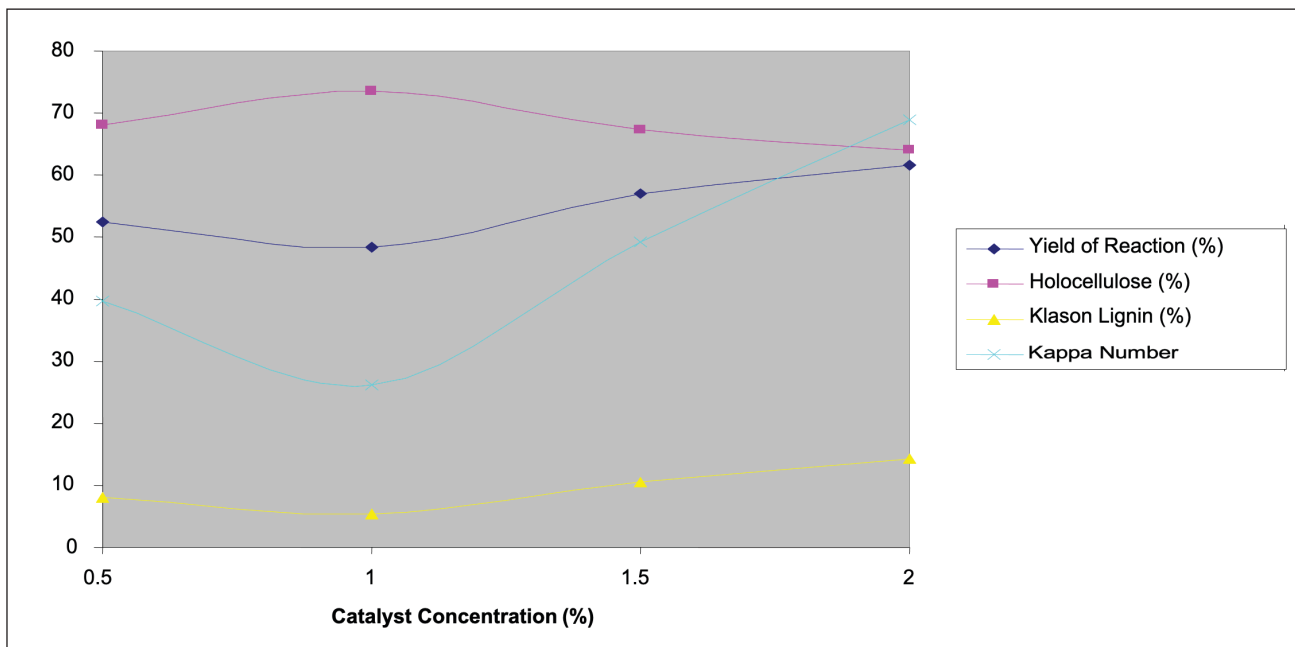
Physical properties of the pulp

Standard handsheets of 100 g/m² were prepared in a laboratory sheet former from the pulps after beating them to 40°SR in a laboratory Valley beater. The paper handsheets were further dewatered by pressing in the lab sheet press and then placing them in a rotary dryer for 24 h at 60°C–70°C. The dried sheets were kept in airtight black polythene bags for subsequent use and analysis of important properties. Before testing physical and optical properties, handsheets were placed in an environment chamber for 3 h, which was maintained at a temperature of 25°C and relative humidity of 52%. The paper handsheets made from rice straw pulp were conditioned and then tested as per TAPPI Standard Test Methods for brightness (T 452 "Brightness of pulp, paper and paperboard"), printing opacity (T 425 "Opacity

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1. Effect of formic acid concentration on pulp yield, holocellulose, Klason lignin, and kappa number.



2. Effect of sulfuric acid (H_2SO_4 catalyst) concentration on pulp yield, holocellulose, Klason lignin, and kappa number.

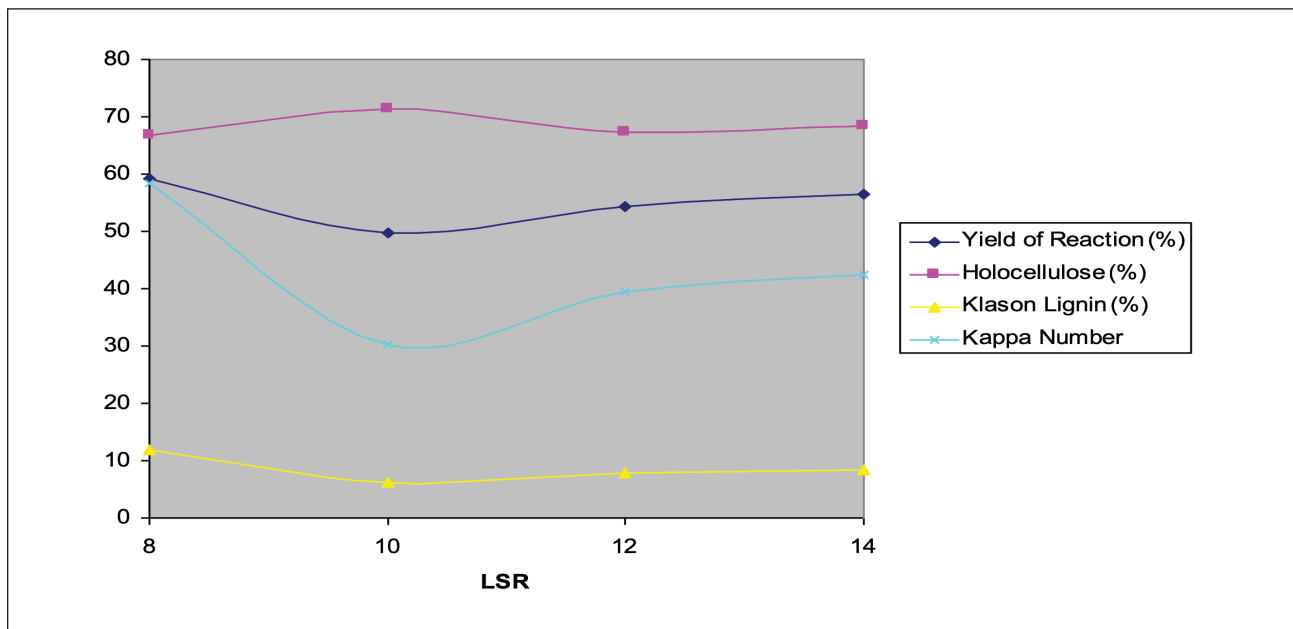
of paper”), burst strength (T 403 “Bursting strength of paper”), tear strength (T 414 “Internal tearing resistance of paper”), and tensile strength (T 494 “Tensile properties of paper and paperboard”).

RESULTS AND DISCUSSION

Effect of concentration of formic acid

The experimental results show a trend for variations in pulp yield percentage; holocellulose content in pulp; Klason lignin percentage in pulp; and kappa number with change of concentration of formic acid in the pulping

reaction. The increase in concentration of formic acid provides better delignification of rice straw and better quality pulp with higher cellulose and lower lignin content. Pulp yield decreases with increase of formic acid concentration in liquor due to an increase in delignification and solubilization of hemicelluloses in formic acid. As high yield is also associated with high lignin percentage in pulp and high kappa number (characterized as very “hard” pulp in the paper industry), the quality of pulp obtained is not suitable for further processing. The optimum quality pulp obtained had a kappa number of 26 when the reaction was



3. Effect of liquor-to-straw ratio (LSR) on pulp yield, holocellulose, Klason lignin, and kappa number.

carried out with 85% acetic acid concentration and 1% catalyst concentration. The kappa number is one of the most important parameters for checking the quality of the final pulp after any pulping process. A kappa number of 20–30 is considered good for papermaking. Higher kappa number values are related to higher residual lignin in pulp, which characterizes the pulp as “hard” pulp that makes refining and further processing to make paper difficult. On the other hand, a lower kappa number (below 20) of pulp results from overcooking and degradation of cellulose and hemicellulose. The pulp with a much lower kappa number will have less fiber strength, and hence poor strength quality in the paper. Therefore, it is required to maintain optimum kappa number in final pulp obtained by any pulping process. The pulping process was maintained at 90°C temperature and 180 min of reaction time; another important process parameter, liquor-to-straw ratio (LSR), was maintained at 10. The detailed effects are shown in **Fig. 1**, as well as the effect of formic acid concentration and selection of the optimum value as per requirements for maximizing pulp yield percentage with high holocellulose content and lower residual lignin percentage in the resulting pulp.

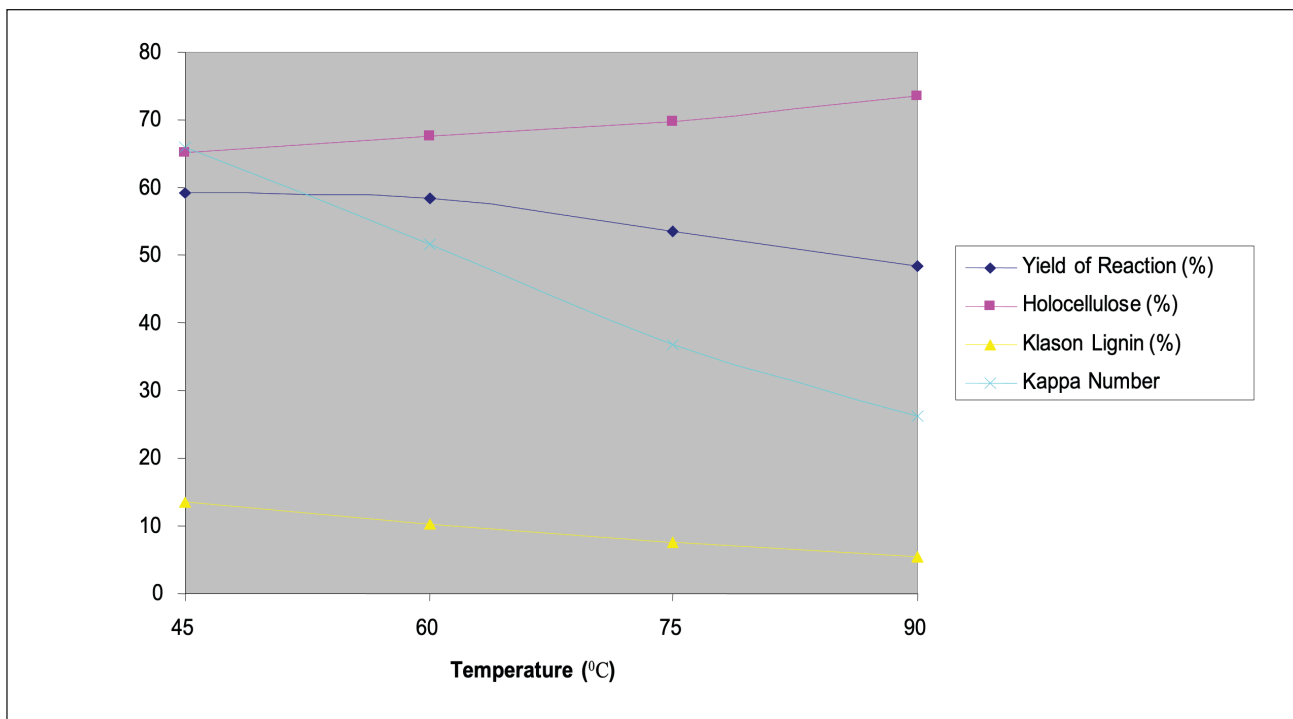
Effect of catalyst concentration

Catalyst (H_2SO_4) concentration of 1% provides the best delignification, with yield reaching to 50%, kappa number approaching 25, and holocellulose percentage in pulp approaching the maximum value of 75% with minimum residual lignin in obtained pulp. The trend of catalyst concentration on pulp yield, holocellulose, Klason lignin, and kappa number are shown in **Fig. 2**, and optimum value is selected as per requirements of the paper

industry, which aims for maximizing the holocellulose percentage in pulp and lowering the residual lignin percentage in the obtained pulp. The study of this trend clearly indicates that the percentage of catalyst in the reaction should be maintained close to 1% to maximize the holocellulose to 75%, and the kappa number obtained trends to a minimum value of 25 (the target is to get kappa number in the range of 25–30), which is very good for production of writing and printing grades of paper. Again, the trend of pulp yield percentage shows that this is approaching 50%, mainly because of maximum removal of lignin and solubilization of hemicellulose at this condition to get the best quality pulp.

Effect of LSR

The amount of liquor present is an important parameter for a uniform and efficient delignification reaction. The whole mass of rice straw (solid phase) should be in contact with another reactant, formic acid, which is present in the liquid phase. Again, the catalyst is in the liquid phase only. The LSR varied between 8 to 14, and the optimum result was obtained at 10. The variation effect is shown in **Fig. 3**. The study of the trend showing the effect of LSR in the formic acid pulping reaction should be maintained at approximately 10 to maximize the holocellulose and to get the best kappa number (which is trending to the optimum value of 25). This kappa number of pulp is very good for production of writing and printing grades of paper. Again the trend is showing that the yield percentage is approaching 50% at LSR equal to 10, which is very good for the chemical pulping process. The pulp yield lowers mainly because of maximization of removal of lignin and solubilization of hemicellulose at this condition to get the best quality of cellulosic pulp.



4. Effect of temperature of reaction on yield, holocellulose, Klason lignin, and kappa number.

Effect of temperature on reaction

The variation of temperature of reaction from 45°C to 90°C has shown a decrease in Klason lignin percentage to nearly 50%. This is verified by observing the decrease of the kappa number of pulp from 65 to 26. As the constituents get degraded in the reaction, the overall yield significantly decreases. The detailed effects are shown in graphs given in **Fig. 4**. This shows the trend for temperature effect on output parameters, primarily the pulp yield percentage, holocellulose in resultant pulp, Klason lignin percentage, and kappa number of pulp. We can select the condition based on our requirement for manufacturing of different paper grades, as some paper requires soft pulp with low kappa number like tissue and some requires hard pulp with high strength like kraft paper. So, this trend will help in selecting the optimum values of reaction input parameters with desired output results.

Effect of time of reaction

The pulping reaction time increase provides a higher extent of completion of the reaction. Time increase up to 180 min shows significant results for lowering of lignin percentage in pulp to a level of 5.7% from 17.3%. A further increase of time does not help significantly in delignification. The change of kappa number also shows similar trends. Detailed effects of time variation are shown in **Fig. 5**. The increase in time taken for the delignification reaction will decrease the pulp production rate of a specific plant and hence will lower the profit (as for any industry, the profit increases with an increase in

production rate). So, the optimum time should be selected for getting the desired kappa number for manufacturing different grades of paper.

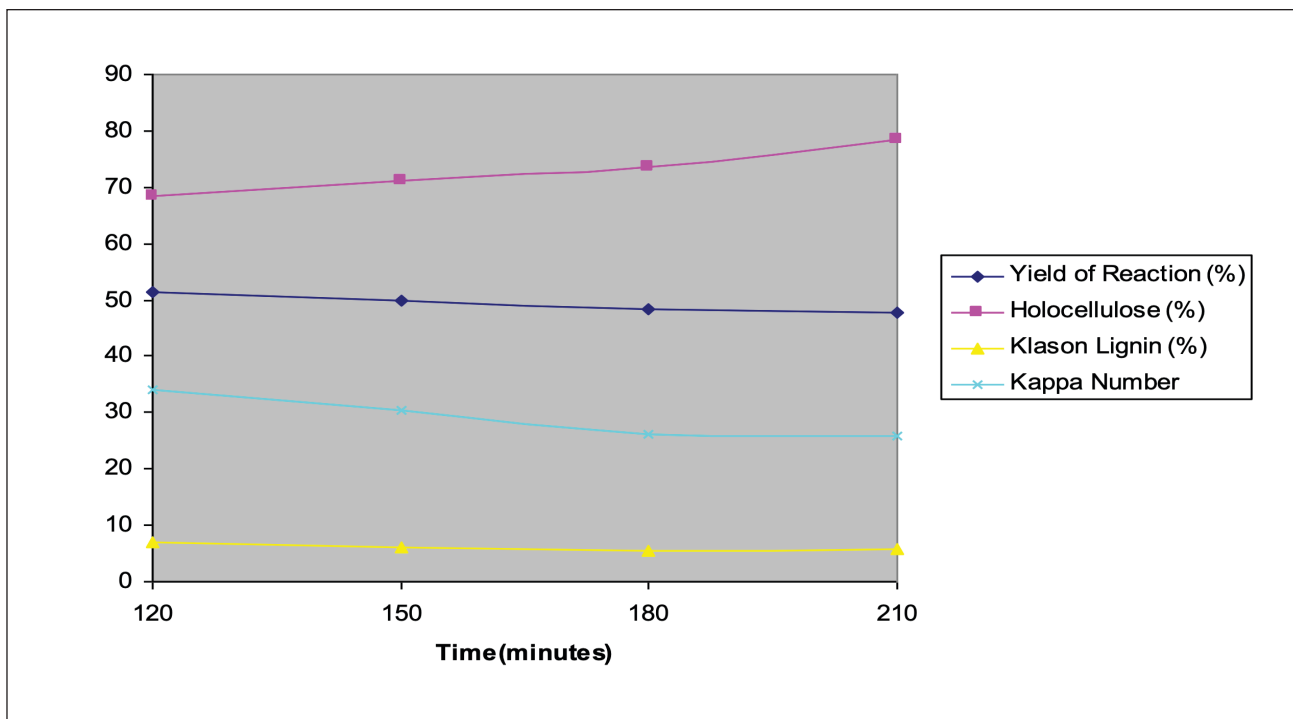
Strength properties

Burst index of paper samples varied from 0.50 to 0.62 kPa.m²/g. The average value of 0.56 kPa.m²/g is suitable for Maplitho grade or newsprint paper. Tear index of paper samples varied from 3.20 to 3.33 mN.m²/g. The average value of 3.28 mN.m²/g is good for average grades of writing and printing paper. Tensile index has values in between 21.0 to 22.20 N.m/g, and the average value is 21.60 N.m/g. The lower value of tensile, tear, and burst index is with nearly 14% filler content in paper, so it is quite good considering such a heavy filler load. Further addition of 10%–30% long fiber (as is the usual practice in for papermaking with short fiber pulp) in rice straw-based short fiber stock will significantly enhance all the strength properties and make it suitable for good quality writing, printing, and speciality papers like laminate base papers, due to better bonding between fibers.

Optical properties

ISO brightness of unbleached pulp was between 24.88% to 26.80% ISO. The brightness obtained for unbleached rice straw pulp is good and can be easily improved by bleaching to 80%–85% ISO brightness using conventional industrial bleaching sequences.

The opacity of paper made with bleached pulp varies between 97.8% to 98.6%. This is very high and makes the



5. Effect of time of reaction on yield, holocellulose, Klason lignin, and kappa number.

paper very good for writing and printing purposes, as readability improves with increase of opacity. This will provide good quality pulp for manufacturing laminate base papers.

SUMMARY

Rice straw has a good potential to be a resourceful raw material for cellulosic fibers and SiO_2 . It may prove to be a major source of natural cellulosic fibers for a number of industrial applications. The very high ash percentage (17.4%) and silica percentage (13.3%) of rice straw in comparison to other conventional raw materials makes it very difficult to be handled by conventional chemical pulping (soda or kraft) processes. Silica present in rice straw is inside the epidermis cells and has a very fine size (nanoscale). The lignin percentage (17.3%) in rice straw is lower than most of the conventional raw materials, so it is easier to delignify. The increase in concentration of formic acid favors the delignification and provides better quality pulp with lower kappa number. Pulp yield decreases with increase of formic acid concentration in liquor due to the increase in delignification and solubilization of hemicelluloses. Catalyst (H_2SO_4) concentration of 1% provides the best delignification, minimum kappa number, and maximum holocellulose percentage in pulp. The LSR maintained at 10 provides a better result in the pulping process. The reaction temperature increase provides a decrease in Klason lignin percentage in the pulp, and hence there is a remarkable decrease in the kappa number of the pulp.

The pulping reaction time increase results in an increase of delignification, but a time increase after 180 min does not add any significant positive change. So, 3 h of time is optimum. Unlike in soda or kraft pulping, the amount of ash and silica retained in the pulp is very high, as ash percentage and silica percentage in the pulp are 15.2 and 13.5, respectively. Weight average fiber length of acetic acid pulp of rice straw is 0.80 mm, which is good enough for manufacturing lower and average grades of paper. The brightness of paper handsheets made with unbleached pulp was approximately 27% ISO, which can be considered good. Printing opacity of acetic acid-delignified pulp was very high (above 98%), which provides very good quality for laminate base, writing, and printing grades of paper. This very high opacity is because of inherent ash (silica) present in the pulp. Again, because the particles are enclosed in epidermis cells and have size less than $1\text{ }\mu\text{m}$, this will not cause any significant friction and damage to machine parts. The addition of fillers like calcium carbonate, titanium dioxide, and silicates is done to get higher opacity and better surface finish in most grades of paper. Therefore, the inherent silica present in pulp will decrease the required quantity of external fillers added. **TJ**

ACKNOWLEDGEMENT

Dr. A.S.K. Sinha is thankful to Mr. Ramnik Aggarwal, senior technician (chemical technology), for his supportive attitude in the laboratories where experimental works were carried out.

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ABOUT THIS PAPER

Cite this article as:

Sinha, A., *TAPPI J.* 20(8): 489(2021).
<https://doi.org/10.32964/TJ20.8.489>

DOI: <https://doi.org/10.32964/TJ20.8.489>

ISSN: 0734-1415

Publisher: TAPPI Press

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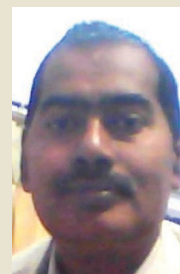
ABOUT THE AUTHORS

The process studied here is based on converting an agricultural waste to fibers containing inherent silica. This will provide a new industrial process and a new fiber resource in the form of rice straw. It differs from previous related work in that past studies were about utilization of coal fly ash as a filler in laminate base papers. The most complicated aspect of this research was that very little work has been done on rice straw for papermaking, so finding suitable pulping process conditions for rice straw was difficult.

Through this study, I found that there is no need to add filler, as a significant percentage (nearly 15%) is present with rice straw fibers. This fiber can also be used for small scale industries due to low temperature and pressure conditions used in the pulping process. The most interesting finding in this research was that formic acid can be used for pulping of rice straw through simple handling procedures

and that paper and board hand-sheets can be manufactured using rice straw fibers.

In the future, paper industries can utilize rice straw as a new fiber resource produced at large scale in a significant number of countries. New grades may be developed due to presence of inherent silica (13%) in the pulp fiber. The next step is a techno-economic analysis with a process design and possibility study for scaling up the process.



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