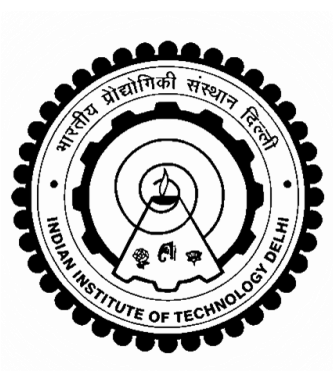


**STUDIES ON EXTRACTION AND APPLICATION OF PEARL
MILLET (*PENNISETUM GLAUCUM*) FIBRES**

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DEPARTMENT OF TEXTILE & FIBRE ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY DELHI

JULY 2023

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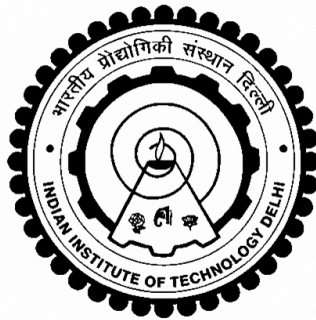
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Submitted

in fulfilment of the requirements of the degree of Doctor of Philosophy

to the



INDIAN INSTITUTE OF TECHNOLOGY DELHI

July 2023

CERTIFICATE

This is to certify that the thesis titled ‘**Studies on Extraction and Application of Pearl Millet (*Pennisetum Glaucum*) Fibres**’, being submitted by Ms Manisha Yadav to the Indian Institute of Technology Delhi, for the award of the degree of Doctor of Philosophy, is a record of bonafide research work carried out by her. She has worked under our guidance and supervision and fulfilled the requirements for submitting the thesis, which has attained the standard required for a PhD degree at this Institute.

The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for the award of any degree or diploma.

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MANISHA YADAV

ABSTRACT

Lignocellulosic biomass is currently being studied worldwide as a source of cellulose for various value-added applications. In this study, the straw of Indian Pearl millet (PMS) (*Pennisetum glaucum*) was systematically studied and characterised for the first time. The feedstock was separated into peripheral leaves, sheath, and core, and their chemical composition was determined. Raw feedstock contains about 46% cellulose, with 52%, 40%, and 44% in the core, sheath, and leaves, respectively. The other components include ~ 24% hemi cellulose, ~17% lignin, and 11% ash. Morphologically, the straw comprises a honeycomb structure made from thin-walled vascular cells. A simple alkali treatment was used to extract pure cellulose from PMS. Extracted cellulose was processed to produce long fibres and short fluff fibres. The crystallinity of long fibers and fluff was 65% and 57%, respectively, and the moisture content for both was 8-9%. The tenacity of long fibres ranged from 180.4-292.86 MPa, and elongation was 2.6-3.8%. The fluff fibres were tested and evaluated as a possible absorbent for use in feminine hygiene products. They show absorption of 10g of water and 11.2g of blood per gram of its own weight.

Twenty commercial sanitary napkins (SN) were compared for physical properties as well as absorbancy of three test fluids. A simple burette-based method was developed to test the absorbancy of samples. SN samples were produced from PM fluff, commercial fluff and a 50:50 mixture of the two. These were tested for absorbancy of DI water, saline and artificial blood. Rewetting and strike-through tests for SN were also carried out. Commercial fluff performed better than PMSF, but the mixture showed much better performance than the commercial fluff on all tests.

The long fibres obtained from PMS were used as a bio sorbent for a cationic dye Methylene Blue (MB) and two anionic dyes Reactive Blue 171 (RB) and Acid Red 57 (AR). Kinetic and thermodynamic studies were conducted to determine the adsorption mechanism of these dyes on PMS fibres. The saturation adsorption of MB was 50 mg/g, while that for anionic dyes was 35 mg/g-38 mg/g. The adsorption process was endothermic in nature. Results indicate that a combination of physical and chemical sorption mechanisms are responsible for the adsorption of dyes on PMS. The study shows that PMS can serve as an indigenous and sustainable source of cellulose for selected industrial applications. By changing the processing sequence, the form and properties of cellulose can be tuned to meet the expected performance requirements.

सार

लिग्नोसेल्यूलोसिक बायोमास का वर्तमान में दुनिया भर में विभिन्न मूल्यवर्धित अनुप्रयोगों के लिए सेलूलोज़ के स्रोत के रूप में अध्ययन किया जा रहा है। इस अध्ययन में, पहली बार भारतीय बाजरा के पुआल (बाकेपु) (पेनिसेटम ग्लौकम) का व्यवस्थित अध्ययन और लक्षणवर्णन किया गया। कच्चे माल को परिधीय पत्तियों, खोल और गूदा में अलग किया गया था और उनकी रासायनिक संरचना निर्धारित की गई थी। कच्चे माल में लगभग 46% सेलूलोज़ होता है, जिसमें क्रमशः गूदा, खोल और पत्तियों में 52%, 40% और 44% होता है। अन्य घटकों में ~ 24% हेमीसेलूलोज़, ~ 17% लिग्निन और 11% राख शामिल हैं। रूपात्मक रूप से, पुआल में पतली दीवार वाली संवहनी कोशिकाओं से बने मधुकोश की संरचना होती है। बाकेपु से शुद्ध सेलूलोज़ निकालने के लिए एक साधारण क्षार उपचार का उपयोग किया गया था। निकाले गए सेल्यूलोज को लंबे रेशे और छोटे फूले हुए रेशे बनाने के लिए संसाधित किया गया था। लंबे रेशे और फूले हुए रेशों की स्फटिकता क्रमशः 65% और 57% थी और दोनों के लिए नमी की मात्रा 8-9% थी। लंबे रेशों की दृढ़ता 180.4-292.86 एम पी एके बीच थी और बढ़ाव 2.6-3.8% था। फूले हुए रेशों का परीक्षण और मूल्यांकन स्त्री स्वच्छता उत्पादों में उपयोग के लिए संभावित शोषक के रूप में किया गया था। वे अपने वजन के प्रतिग्राह 10 ग्राम पानी और 11.2 ग्राम रक्त का अवशोषण दिखाते हैं।

बीस व्यावसायिक आरोग्य कर रुमाल (आरु) की तुलना भौतिक गुणों के साथ-साथ तीन परीक्षण तरल पदार्थों के अवशोषण के लिए की गई थी। नमूनों की अवशोषण क्षमता का परीक्षण करने के लिए एक सरल ब्यूरेट-आधारित विधि विकसित की गई थी। आरु नमूने पीएम फूले हुए रेशे, व्यावसायिक फूले हुए रेशे और दोनों के 50:50 मिश्रण से तैयार किए गए थे। इनका डी आई पानी, खारा और कृत्रिम रक्त के अवशोषण के लिए परीक्षण किया गया था। आरु के लिए पुनः गीला करना और स्ट्राइक थ्रूपरीक्षण भी किए गए। बाकेपु की तुलना में व्यावसायिक फूले हुए रेशों ने बेहतर प्रदर्शन किया, लेकिन मिश्रण सभी परीक्षणों पर व्यावसायिक फूले हुए रेशों की तुलना में बेहतर प्रदर्शन दिखाता है।

बाकेपु से प्राप्त लंबे रेशों का उपयोग एक धनायनित डाई मेथिलीन ब्लू (एमबी) और दो ऋणायनी डाई रिएक्टिव ब्लू 171 (आरबी) और एसिड रेड 57 (एआर) के लिए जैवशर्बत के रूप में किया गया था। बाकेपु पर इन रंगों के सोखने के तंत्र को निर्धारित करने के लिए गतिज और ऊष्म प्रवैगि की अध्ययन किए गए। एमबी का संतृप्ति सोखना 50 मिलीग्राम/ग्राम था, जबकि ऋणायनी रंगों के लिए यह 35 मिलीग्राम/ग्राम- 38 मिलीग्राम/ग्राम था। सोखने की प्रक्रिया प्रकृति में ऊष्मा शोषी थी। परिणाम इंगित करते हैं कि बाकेपु रंगों के सोखने के लिए भौतिक और रासायनिक सोखना तंत्र का संयोजन जिम्मेदार है। अध्ययन से पता चलता है कि बाकेपु धनायनित औद्योगिक अनुप्रयोगों के लिए सेल्यूलोज के

एक स्वदेशी और स्थायी स्रोत के रूप में काम कर सकता है। प्रसंस्करण अनुक्रम को बदल कर, अपेक्षित प्रदर्शन आवश्यकताओं को पूरा करने के लिए सेलूलोज़ के रूप और गुणों को परिवर्तित किया जा सकता है।

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LIST OF ABBREVIATIONS

PM	Pearl Millet
PMS	Pearl Millet Straw
PMF	Pearl Millet Fluff
PMSF	Pearl Millet Straw Fibres
PMLF	Pearl Millet long fibres
PMSC	Pearl Millet Single Cell
RCR	Residue to Crop Ratio
MCC	Micro Crystalline Cellulose
NCC	Nano Crystalline Cellulose
NF	Nitrogen Factor
ODW	Oven Dry Weight
XRD	X- Ray Diffraction
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscope
TGA/DTG	Thermo Gravimetric Analysis/Derivative Thermogravimetry
SCB	Sugarcane Bagasse
SSB	Sweet Sorghum Bagasse
HPLC	High Performance Liquid Chromatography
NREL	National Renewable Energy Laboratory
NF	Nitrogen Factor
ODW	Oven Dry Weight
LC	Lignocellulosic
LCB	Lignocellulosic Biomass
PMLF	Pearl Millet long fibres
SMC	Super mass colloidier
WHC	Water Holding Capacity
MR	Moisture Regain
WOR	Work of Rupture
SN	Sanitary Napkin
AUL	Absorbancy Under Load
NGOs	Non- Government Agencies
DI	Deionized
MDF	Medium Density Fibreboard
SP	Smallest Pore
MFP	Mean Flow Pore
NWSP	Nonwoven Standard Procedures
PP	Polypropylene
SAP	Super Absorbent Polymer
AD	Aerial Density
GSM	Grams per Square Meter
PSD	Pore Size Distribution
MB	Methylene Blue

RB 171	Reactive Blue 171
AR 57	Acid Red 57
SDG	Sustainable Development Goals