

**DEVELOPING NON-WOOD PAPER AND OPTIMIZING FOR RURAL
SCALE PACKAGING**

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DEVELOPING NON-WOOD PAPER AND OPTIMIZING FOR RURAL SCALE PACKAGING

by

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CERTIFICATE

This is to certify that the thesis titled “**Developing Non-wood Paper and Optimizing for Rural Scale Packaging**” being submitted by **Mr. Vinayak Dattatray Fasake** (2017RDZ8226) 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 him. He has worked under my guidance and supervision and has fulfilled the requirements for the submission of this thesis, which has attained the standard required for a Ph.D. degree at this Institute. The results represented in this thesis have not been submitted elsewhere for the award of any degree or diploma.

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ABSTRACT

The Indian subcontinent is blessed with a large cattle population of around 535.82 million and rearing cattle had been an integral part of our civilization. Cattle dung is a source of significant raw material for industries like Agri, pulp and paper, bioprocessing of resources to wealth and energy. The demand of cow dung as a rural resource has been increasing as we realize its potential to replace conventional resources. One such industry is the pulp and paper industry where the fiber extracted from cow dung can be an effective raw material for packaging paper. Cattle dung has a special significance in India and had never been considered a waste material. The present study was undertaken with three key objectives to separate the undigested raw materials (raw dung fibers) from fresh cattle dung and test them for different physiochemical properties. Three types of undigested animal dung fibers from an Indigenous cow (IDF), Jersey cow (JDF), and Buffalo (BDF) were taken. Wheat straw (WS) was the main diet of all animals. The cellulose, hemicellulose, and lignin content for all animal dung samples were found in a range of (29%-31.50%), (21%-23.50%), and (11%-13%), respectively. Scanning electron microscopy (SEM) analysis shows the layer matrix, skeleton structure, and honey-comb-like packed arrangement of fiber making. The abundant holocellulose and low lignin and ash contents were observed in all samples. Based on the results and considering large numbers of the animal population, indigenous cow dung was considered for further study.

In the second objective of the study, the raw dung fibers (RDF) separated from fresh cow dung and digested dung fibers (DDF) were collected from an anaerobically digested bottled biogas plant. The entire study was planned to characterize and analyze both RDF and DDF samples. The results showed that the cellulose and hemicellulose content was in the range of 36%-38% and 21%-23%, respectively. The total lignin contents in RDF and DDF were 26.17% and 31.92%, while the ash content of 6.2% was found in DDF followed by 5.8% in RDF. Surface

characteristics of RDF and DDF were investigated using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The proximate analysis and morphological characteristics of both samples showed that dung fibers are sustainable and potential non-wood material for various industrial applications. Also, extracting undigested particles from raw cattle dung is a convenient method to separate RDF.

The use of biomass by removing lignin and hemicellulose for paper production and packaging has become a subject of current attention due to the increased demand for green energy content and environmental protection. Another objective of this research was to optimize the delignification parameters for RDF by using a central composite design (CCD). In this objective, the effect of independent factors viz. temperature and time on dependent variables viz. yield, cellulose, hemicellulose and lignin of dung fiber were investigated. The optimum conditions to produce maximum yield ($78.15 \pm 1.5\%$), cellulose ($48.08 \pm 0.96\%$), and lignin ($30.05 \pm 1.83\%$) were obtained at 160°C , cooking time 5 min, and solid to-water ratio 1:10. The paper sheet was prepared by using hydrothermally treated (HT) dung material and an additional blending material (paddy straw pulp and waste cotton rag material pulp) for various proportions. The paper was tested for physical and mechanical properties. Results show that (C50) a blend of 50% of dung fiber in waste cotton rag pulp exhibits maximum tensile and bursting index. The main advantage of the proposed processing scheme is found to be environment friendly and it valorizes the hemicellulose fraction of dung fiber and also exhibited more yield.

The chemically treated (NaOH) cattle dung pulp parameters were optimized using RSM. The cellulose, hemicellulose, and lignin were considered variables affecting the final properties of the prepared paper in the experimental design. In the selected treatment of 3% NaOH at 171°C for 15 min, the percentage reduction of lignin is 96.17% and retained almost 50% of holocellulose in dung fibers, which prevented excessive carbohydrate breakdown. Such results exhibit the

quadratic model proved to be more accurate. The chemical percentage and holding temperature were key factors in determining the total pulp yield. The relative errors between predicted and actual experimental results were less than 5%, indicating that the model used to show the correlation between variables and response was correct. The tensile strength was slightly increased in C20 as compared to C10 and then decreased with the increase in dung fiber pulp content. Vice versa, the bursting strength continuously increased from C10 to C30 due to the compact structure and better interaction between the dung fibers and cotton rag pulp. The blending percentage should be altered based on our end-use application, which can aid in the production of high-quality paper.

सारांश

भारतीय उपमहाद्वीप लगभग 535.82 मिलियन मवेशियों की एक बड़ी आबादी के साथ समृद्ध है, और मवेशियों का पालन हमारी सभ्यता का एक अभिन्न अंग था। मवेशियों का गोबर, कृषि, लुगदी और कागज, संसाधनों से धन और ऊर्जा के लिए जैव प्रसंस्करण जैसे उद्योगों के लिए महत्वपूर्ण कच्चे माल का स्रोत है। ग्रामीण संसाधन के रूप में गोबर की मांग बढ़ रही है क्योंकि हम पारंपरिक संसाधनों को बदलने की इसकी क्षमता की अनुभूति करते हैं। लुगदी और कागज उद्योग में ऐसा उद्योग जहां गाय के गोबर से निकाले गए फाइबर को कागज की पैकेजिंग के लिए प्रभावी रूप से कच्चा माल बनाया जा सकता है। भारत में मवेशियों के गोबर का एक विशेष महत्व है और इसे कभी भी अपशिष्ट पदार्थ नहीं माना जाता है। वर्तमान अध्ययन तीन प्रमुख उद्देश्यों के साथ किया गया था ताकि ताजा मवेशियों के गोबर से बिना पचे कच्चे माल (कच्चे गोबर के फाइबर) को अलग किया जा सके और उन्हें विभिन्न भौतिक रासायनिक गुणों के लिए परीक्षण किया जा सके। एक स्वदेशी गाय (आईडीएफ), जर्सी गाय (जेडीएफ), और भेंस (बीडीएफ) से तीन प्रकार के बिना पचे हुए पशु गोबर फाइबर लिए गए थे। गेहूं का भूसा (डब्ल्यूएस) सभी जानवरों का मुख्य आहार था। सभी जानवरों के गोबर के नमूनों के लिए सेल्यूलोज, हेमिकेलुलोज और लिग्निन सामग्री क्रमशः (29-31.50%), (21-23.50%), और (11-13%) की सीमा में पाई गई। स्कैनिंग इलेक्ट्रॉन माइक्रोस्कोपी (एसईएम) विश्लेषण, फाइबर बनाने की परत मैट्रिक्स, कंकाल संरचना और शहद-कंधी जैसी पैक व्यवस्था को दर्शाता है। सभी नमूनों में प्रचुर मात्रा में होलोसेल्यूलोज और कम लिग्निन और राख सामग्री देखी गई। परिणामों के आधार पर और जानवरों की आबादी में बड़ी संख्या में योगदान देने के लिए, स्वदेशी गोबर को आगे के अध्ययन के लिए विचार किया गया था।

अध्ययन के दूसरे उद्देश्य में, ताजा गाय के गोबर से अलग कच्चे गोबर फाइबर (आरडीएफ) और पचने वाले गोबर फाइबर (डीडीएफ) को अवायवीय रूप से पचने वाले बोटलबंद बायोगैस संयंत्र से एकत्र किया गया था। पूरे अध्ययन को आरडीएफ और डीडीएफ नमूनों दोनों को चिह्नित करने और विश्लेषण करने की योजना बनाई गई थी। परिणामों से पता चला है कि सेल्यूलोज और हेमिकेलुलोज सामग्री क्रमशः 36-38% और 21-23% की सीमा में है। आरडीएफ और डीडीएफ में कुल लिग्निन सामग्री 26.17% और 31.92% थी, जबकि डीडीएफ में 6.2% की राख सामग्री पाई गई, जिसके बाद आरडीएफ में 5.8% थी। आरडीएफ और डीडीएफ की सतह विशेषताओं की जांच स्कैनिंग इलेक्ट्रॉन माइक्रोस्कोपी (एसईएम) और फूरियर ट्रांसफॉर्म इन्फ्रारेड स्पेक्ट्रोस्कोपी (एफटीआईआर) का उपयोग करके की गई थी। दोनों नमूनों के अनुमानित विश्लेषण और रूपात्मक विशेषताओं से पता चला है कि गोबर फाइबर विभिन्न औद्योगिक अनुप्रयोगों के लिए एक स्थायी संभावित गैर-लकड़ी सामग्री है। अंत में, लेखकों ने पाया कि कच्चे मवेशियों के गोबर से बिना पचे हुए कणों को निकालना कच्चे गोबर फाइबर (आरडीएफ) को अलग करने का एक सुविधाजनक तरीका है।

कागज उत्पादन और पैकेजिंग के लिए लिग्निन और हेमिकेलुलोज को हटाकर बायोमास का उपयोग हरित ऊर्जा सामग्री और पर्यावरण संरक्षण की बढ़ती मांग के कारण वर्तमान ध्यान का विषय बन गया है। इस शोध का एक अन्य उद्देश्य एक केंद्रीय समग्र डिजाइन (सीसीडी) का उपयोग करके आरडीएफ के लिए डेलिगेनिफिकेशन मापदंडों को अनुकूलित करना था। इस पेपर में, स्वतंत्र कारकों जैसे तापमान और समय के निर्भर चर पर प्रभाव की जांच की गई थी जैसे उपज, सेल्यूलोज हेमिकेलुलोज, और गोबर फाइबर के लिग्निन। अधिकतम उपज ($78.15 \pm 1.5\%$), सेल्यूलोज ($48.08 \pm 0.96\%$), और लिग्निन ($30.05 \pm 1.83\%$) का उत्पादन करने के लिए इष्टतम परिस्थितियां 160 डिग्री सेल्सियस, खाना पकाने का समय 5 मिनट, और ठोस से पानी का अनुपात 1:10 पर प्राप्त किया जाता है। पेपर शीट को हाइड्रोथर्मल रूप से उपचारित (एचटी) गोबर सामग्री और विभिन्न अनुपातों के लिए एक अतिरिक्त सम्मिश्रण सामग्री (धान की पुआल का गूदा और अपशिष्ट कपास कचरा सामग्री लुगदी) का उपयोग करके तैयार किया गया था। पेपर को भौतिक और यांत्रिक गुणों के लिए परीक्षण किया गया था। परिणाम बताते हैं कि (सी 50) अपशिष्ट कपास के कचरे में 50% गोबर फाइबर का मिश्रण अधिकतम तन्यता और बीयूआरस्टिंग इंडेक्स प्रदर्शित करता है। प्रस्तावित प्रसंस्करण योजना का मुख्य लाभ पर्यावरण के अनुकूल है, गोबर फाइबर के हेमिकेलुलोज अंश को महत्व देता है, और अधिक उपज का प्रदर्शन करता है।

रासायनिक रूप से उपचारित (एनएओएच) मवेशियों के गोबर के गूदे के मापदंडों को आरएसएम का उपयोग करके अनुकूलित किया गया था। सेल्यूलोज, हेमिकेलुलोज और लिग्निन को प्रयोगात्मक डिजाइन में तैयार पेपर के अंतिम गुणों को प्रभावित करने वाले चर के रूप में माना जाता था। 15 मिनट के लिए 171 डिग्री सेल्सियस पर 3% एनएओएच के चयनित उपचार में, लिग्निन की प्रतिशत कमी 96.17% है और गोबर फाइबर में होलोसेल्यूलोज का लगभग 50% बरकरार है, जिसने अत्यधिक कार्बोहाइड्रेट टूटने को रोका। इस अध्ययन में, द्विघात मॉडल अधिक सटीक साबित हुआ। कुल लुगदी उपज निर्धारित करने में रासायनिक प्रतिशत और धारण तापमान महत्वपूर्ण कारक थे। अनुमानित और वास्तविक प्रयोगात्मक परिणामों के बीच सापेक्ष त्रुटियां 5% से कम थीं, यह दर्शाता है कि चर और प्रतिक्रिया के बीच सहसंबंध दिखाने के लिए उपयोग किया जाने वाला मॉडल सही था। सी-10 की तुलना में सी-20 में तन्यता शक्ति थोड़ी बढ़ गई थी और फिर गोबर फाइबर लुगदी सामग्री में वृद्धि के साथ कम हो गई थी। इसके विपरीत, कॉम्पैक्ट संरचना और गोबर फाइबर और कपास के गूदे के बीच बेहतर बातचीत के कारण फटने की ताकत लगातार सी-10 से सी-30 तक बढ़ गई। सम्मिश्रण प्रतिशत को हमारे अंतिम उपयोग अनुप्रयोग के आधार पर बदल दिया जाना चाहिए जो उच्च गुणवत्ता वाले कागज के उत्पादन में सहायता कर सकता है।

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LIST OF ABBREVIATIONS

Bi	Bismuth
BSS	British Standard Sieve
BDF	Buffalo Dung Fiber
Ca	Calcium
OH	Carbon-Hydrogen bond
cm	Centimeter
CCD	Central Composite Designs
CRDT	Centre for Rural Development and Technology
Cr	Chromium
CNG	Compressed natural gas
°C	Degree Celsius
EDX	Energy Dispersive X-Ray
FTIR	Fourier Transform Infrared Spectroscopy
GOBAR	Galvanizing Organic Bio-Agro Resources
g	Gram
HPLC	High Performance Liquid Chromatography
h	Hour
Kg	Kilogram
OH	Hydroxide
IS	Indian Standard
IDF	Indigenous Dung Fibers
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
JDF	Jersey Dung Fibers
kV	Kilovolt
Pb	Lead
Mn	Manganese
Hg	Mercury

µg	Micro gram
µm	Micro metre
µS/cm	microsiemens per centimeter
mg/L	milligrams per liter
ml	Millilitre
mm	Millimeters
MT	Million tones
min	Minutes
Mo	Molybdenum
NREL	National Renewable Energy Laboratory
%	Percent
P	Phosphorus
K	Potassium
RSM	Response Surface Methodology
rpm	Revolutions per minute
SEM	Scanning Electron Microscopy
Si	Silicon
NaOH	Sodium Hydroxide
SAS	Statistical Analysis Software
TNAU	Tamil Nadu Agricultural University
TAPPI	Technical Association of the Pulp and Paper Industry
V	Vanadium
WS	Wheat straw