# “PROCESSING AND PERFORMANCE PROPERTIES OF POLYPROPYLENE/SISAL FIBRE COMPOSITES AND THEIR FOAMS”

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**DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING**

**INSTITUTE OF TECHNOLOGY DELHI**

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*by*


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**DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING**


***Submitted***

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

***to the***



**INDIAN INSTITUTE OF TECHNOLOGY DELHI**

**MAY 2023**

*Dedicated to my*

*Father "Baba V. Bhagat"*

*&*

*Mother "Sheela Bhagat"*

# Certificate

This is to certify that the thesis entitled **"Processing and Properties of Polypropylene/Sisal fibre composites and their foam"** being submitted by **Mr. Ajit Babarao Bhagat** to the **Indian Institute of Technology Delhi,** for the fulfilment of award of the degree **"Doctor of philosophy"** is a record of bonafide research work carried by him under our supervision and guidance. This thesis has been prepared in conformity with the rules and regulations of the Indian Institute of Technology Delhi, New Delhi.

We further certify that the thesis has attained a standard required for the Ph.D. degree of the institute. To the best of our knowledge, the research reported and result presented in the thesis have not been submitted in part or full to any other institute or university for the award of any other degree or diploma.

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# Abstract

This work explores the processability and foamability of Polypropylene/sisal fibre (PP/SF) composites having near critical fibre length and short fibres. The addition of near critical fibre length is expected to cause the rheological percolation which is not possible in short fibre composites. Sisal fibres were treated with sodium bicarbonate ($NaHCO_3$) for different time intervals and the optimization of the treatment time was done by analysing fibre roughness, crystallinity and tenacity. Specifically, the effect of fibre treatment, fibre length, and addition of compatibilizer (polypropylene grafted maleic anhydride) on the performance properties of the composites were studied. The critical fibre length for the PP/SF composite system was found to be 8 mm. PP/SF composites were prepared using an internal mixer by varying fibre concentration (10, 20, 30, 40 wt.%), and the average fibre length (~17 mm) in the composites confirms the presence of near-critical fibre length. The investigation of the performance of PP/SF composites having near-critical fibre length was carried out using physicomechanical, thermal, rheological, and dynamic mechanical parameters. With increase in fibre concentration, significant improvements in mechanical and thermal properties were observed. The results show that sisal fibre has an undeniable impact on the dynamic mechanical properties of the prepared composites as the storage modulus and loss modulus increase with increase in sisal fibre concentration. The improvement in modulus results from the physical interaction between PP and sisal fibre; however, the load transfers from fibre to matrix restrict the segmental mobility of the PP chains. Shear rheology analysis confirms strong shear thinning behaviour as the power-law exponent decreases with increased fibre concertation. However, marginal improvement in the mechanical properties were observed in case of PP/SF composites with short fibres. The marginal improvement in the properties might be due to the short length of fibres, which is unable to transfer load from fibre to matrix.

Further, the foamability of PP and the prepared composites (having both NCFL and short fibre) were carried out with batch foaming setup using supercritical $CO_2$ (sc-$CO_2$) as a physical blowing agent. Foam processing was performed at different temperatures (145, 155, and 165ºC); however, the optimized foaming temperature was found to be 145ºC. PP/SF short fibres composites were foamed at the optimized temperature. The incorporation of sisal fibre into the PP matrix helps in cell nucleation; hence, cell density increases and cell size decreases, as analysed from SEM. Effect of fibre length on the foamability of PP/SF composites were observed using SEM and the results revel that the microcellular structure being present in NCFL based PP/SF composites. The prepared rigid microcellular foams offer properties that can be potentially used to produce high strength-to-weight ratio structural components, which could be employed in applications related to sound absorption.

The continuous foam processability of polypropylene/Sisal fibre composites were determined using extrusion foaming. $CO_2$ gas was used as a physical blowing agent. Foaming was carried out at 60 Bar pressure, screw speed of 40 rpm, and at different temperatures (160ºC and 200ºC). The lowest density and highest volume expansion ratio were found in foams prepared at a temperature of 160ºC. Further, scanning electron microscopy analysis supports the physical property results, and the smallest cell size and more significant cell density were found in the foams prepared at 160ºC. Though cell size increased with fibre concentration (0-10 wt.% of sisal fibre), the microcellular foam structure was found in foams prepared at 160ºC. Thus, it can be concluded that 160ºC was the optimized temperature for foam processing. The addition of sisal fibres in the PP matrix apparently decreases the impact absorb energy, but the foam prepared at 160ºC shows enhanced energy absorption than that of the sample prepared at 200ºC. The prepared rigid foam composites might find the potential application in automobiles (e-vehicles) and packaging.

# सार

यह काम पॉलीप्रोपाइलीन / सिसल फाइबर (पीपी / एसएफ) कंपोजिट की प्रक्रियात्मकता और फोमबिलिटी की पड़ताल करता है, जिसमें महत्वपूर्ण फाइबर लंबाई और छोटे फाइबर होते हैं। लगभग महत्वपूर्ण फाइबर लंबाई के अलावा रियोलॉजिकल परकोलेशन का कारण बनने की उम्मीद है जो छोटे फाइबर कंपोजिट में संभव नहीं है। अलग-अलग समय अंतराल के लिए सिसल फाइबर को सोडियम बाइकार्बोनेट ($NaHCO_3$) के साथ इलाज किया गया था और फाइबर खुरदरापन, क्रिस्टलीयता और दृढ़ता का विश्लेषण करके उपचार समय का अनुकूलन किया गया था। विशेष रूप से, कंपोजिट के प्रदर्शन गुणों पर फाइबर उपचार, फाइबर लंबाई, और कॉम्पिटिबिलाइज़र (पॉलीप्रोपाइलीन ग्राफ्टेड मेनिक एनहाइड्राइड) के प्रभाव का अध्ययन किया गया। PP/SF समग्र प्रणाली के लिए महत्वपूर्ण फाइबर की लंबाई 8 मिमी पाई गई। पीपी/एसएफ कंपोजिट्स को अलग-अलग फाइबर कंसंट्रेशन (10, 20, 30, 40 वजन%) द्वारा एक आंतरिक मिक्सर का उपयोग करके तैयार किया गया था, और कंपोजिट में औसत फाइबर लंबाई (~ 17 मिमी) निकट-महत्वपूर्ण फाइबर लंबाई की उपस्थिति की पुष्टि करती है। निकट-महत्वपूर्ण फाइबर लंबाई वाले पीपी/एसएफ कंपोजिट के प्रदर्शन की जांच फिजियोमेकेनिकल, थर्मल, रियोलॉजिकल और डायनेमिक मैकेनिकल पैरामीटर का उपयोग करके की गई थी। फाइबर एकाग्रता में वृद्धि के साथ, यांत्रिक और तापीय गुणों में महत्वपूर्ण सुधार देखा गया। परिणाम बताते हैं कि सिसल फाइबर का तैयार कंपोजिट के गतिशील यांत्रिक गुणों पर एक निर्विवाद

प्रभाव पड़ता है क्योंकि भंडारण मापांक और हानि मापांक में वृद्धि के साथ सिसल फाइबर एकाग्रता में वृद्धि होती है। पीपी और सिसल फाइबर के बीच भौतिक संपर्क से मापांक में सुधार; हालाँकि, लोड फाइबर से मैट्रिक्स में स्थानांतरित होता है, पीपी श्रृंखलाओं की खंडीय गतिशीलता को प्रतिबंधित करता है। कतरनी रियोलॉजी विश्लेषण मजबूत कतरनी पतले व्यवहार की पुष्टि करता है क्योंकि शक्ति-कानून प्रतिपादक बढ़े हुए फाइबर संघनन के साथ घटता है। हालांकि, छोटे फाइबर वाले पीपी/एसएफ कंपोजिट के मामले में यांत्रिक गुणों में मामूली सुधार देखा गया। गुणों में मामूली सुधार फाइबर की कम लंबाई के कारण हो सकता है, जो फाइबर से मैट्रिक्स में भार स्थानांतरित करने में असमर्थ है।

इसके अलावा, भौतिक ब्लोइंग एजेंट के रूप में सुपरक्रिटिकल सीओ2 (एससी-सीओ2) का उपयोग करके बैच फोमिंग सेटअप के साथ पीपी और तैयार कंपोजिट (एनसीएफएल और शॉर्ट फाइबर दोनों) की फोमबिलिटी की गई। फोम प्रसंस्करण विभिन्न तापमानों (145, 155, और 165 $^{o}$C) पर किया गया था; हालाँकि, अनुकूलित फोमिंग तापमान 145$^{o}$C पाया गया। पीपी/एसएफ शॉर्ट फाइबर कंपोजिट को अनुकूलित तापमान पर फोम किया गया। पीपी मैट्रिक्स में सिसल फाइबर का समावेश सेल न्यूक्लिएशन में मदद करता है; इसलिए, सेल घनत्व बढ़ता है और सेल का आकार घटता है, जैसा कि एसईएम से विश्लेषण किया गया है। PP/SF सम्मिश्र की झाग क्षमता पर फाइबर की लंबाई का प्रभाव SEM का उपयोग करके देखा गया और परिणाम बताते हैं कि NCFL आधारित PP/SF सम्मिश्र में माइक्रोसेलुलर संरचना मौजूद है। तैयार कठोर माइक्रोसेलुलर

फोम गुण प्रदान करते हैं जिनका संभावित रूप से उच्च शक्ति-से-वजन अनुपात संरचनात्मक घटकों का उत्पादन करने के लिए उपयोग किया जा सकता है, जिन्हें ध्वनि अवशोषण से संबंधित अनुप्रयोगों में नियोजित किया जा सकता है।

एक्सट्रूज़न फोमिंग का उपयोग करके पॉलीप्रोपाइलीन / सिसल फाइबर कंपोजिट की निरंतर फोम प्रक्रियाशीलता निर्धारित की गई थी। $CO_2$ गैस का उपयोग भौतिक ब्लोइंग एजेंट के रूप में किया जाता था। फोमिंग 60 बार दबाव, 40 आरपीएम की पेंच गति और विभिन्न तापमानों (160°C और 200°C) पर किया गया था। 160°C के तापमान पर तैयार फोम में सबसे कम घनत्व और उच्चतम आयतन विस्तार अनुपात पाया गया। इसके अलावा, स्कैनिंग इलेक्ट्रॉन माइक्रोस्कोपी विश्लेषण भौतिक संपत्ति के परिणामों का समर्थन करता है, और सबसे छोटा सेल आकार और अधिक महत्वपूर्ण सेल घनत्व 160°C पर तैयार फोम में पाया गया। यद्यपि कोशिका का आकार फाइबर सांद्रता (0-10 सिसल फाइबर का वजन%) के साथ बढ़ गया, 160°C पर तैयार फोम में माइक्रोसेलुलर फोम संरचना पाई गई। इस प्रकार, यह निष्कर्ष निकाला जा सकता है कि फोम प्रसंस्करण के लिए 160°C इष्टतम तापमान था। पीपी मैट्रिक्स में सिसल फाइबर के अलावा प्रभाव अवशोषित ऊर्जा को स्पष्ट रूप से कम कर देता है, लेकिन 160°C पर तैयार फोम 200°C पर तैयार किए गए नमूने की तुलना में ऊर्जा अवशोषण को बढ़ाता है। तैयार कठोर फोम कंपोजिट ऑटोमोबाइल (ई-वाहन) और पैकेजिंग में संभावित अनुप्रयोग पा सकते हैं।

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