

STUDIES IN FUNCTIONALIZATION OF TEXTILES USING MICROENCAPSULATED MATERIALS

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STUDIES IN FUNCTIONALIZATION OF TEXTILES USING MICROENCAPSULATED MATERIALS

by

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CERTIFICATE

This is to certify that the thesis titled ‘Studies in Functionalization of Textiles using Microencapsulated Materials’, being submitted by Mr. Nagender Singh 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 fulfilled the requirements for submission of the thesis which has attained the standard required for a PhD degree of 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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ABSTRACT

In the 21st century, the occurrence of deadly viruses, mosquito-borne and pathogenic diseases have increased significantly. One of the best strategies to avoid the spread of these diseases is to use protective textile products such as functional apparels (antibacterial, antioxidant, antiviral, mosquito repellent), functional mask, scarf, gowns, curtains and bedsheets. New strategies are being explored for the functionalization of textiles by finding alternatives to highly toxic and synthetic chemicals. Since ancient times, essential oils (EOs) are being used for medicinal propose because of their inherent biological properties. However, the use of essential oils (EOs) for the functionalization of textiles is limited owing to their volatile nature. Several attempts have been made to enhance their durability to attain excellent pharmaceutical effects through the textiles. The present research revolves around the development of multifunctional textiles using microencapsulated novel essential oil (EOs) in functional polymers.

In the first part of this research, an attempt was made to develop *in-situ* synthesized chitosan-phosphate microcapsules loaded with thyme oil. Seventeen runs were performed to optimize the polymer-to-oil concentration by measuring encapsulation yield and encapsulation efficiency of the microcapsules. The obtained microcapsules offered more than 71% encapsulation efficiency for all the runs, which was quite significant. The developed microcapsules were applied to the linen fabric using an acrylic binder to impart functional properties. The microcapsules were spherical and owned a matrix type of morphology. Interestingly, the oil release from the microcapsules was found to be governed by diffusion and swelling mechanism. The results were impressive, which showed the presence of oil on the fabric even after 20 washes. The EOs are highly flammable; however, this research attempted to prepare the flame retardant microcapsules loaded with thyme oil. The finished fabric exhibited excellent mosquito repellency (100 %), antioxidant activity (>96 %), pleasant aroma

and moderate flame retardancy. The finished fabric also exhibited excellent antibacterial activity even after repeated launderings. The marginal change was observed in the mechanical properties and air permeability of the fabric. Another attempt was made to confirm the authenticity of this research by changing the EOs (rosemary essential oil). Moreover, the results showed similar trends as observed in the previous section. These results motivate us to explore the new polymers and oils to enhance the microcapsules' encapsulation efficiency and achieve better functional properties.

In the second part of the research, an attempt was made to explore the effect of polymer mixture on the encapsulation efficiency and release kinetics of the microcapsules. The microcapsules were developed using a mixture of chitosan-gelatin as a shell material and rosemary oil as a core material. The prepared microcapsules showed matrix and reservoir type of morphology. The microcapsules offered a sustained oil release profile, which was governed by a diffusion mechanism. The obtained microcapsules offered more than 68 % encapsulation efficiency. The finished fabric showed excellent mosquito repellency, antibacterial activity, antioxidant activity and a pleasant aroma even after repeated launderings. Another attempt was made to confirm the authenticity of the research by changing the EO (Cinnamon bark oil). The obtained microcapsules offered a similar kind of morphology and released mechanism as found in a previous section of the second part of the research. However, the microcapsules offered better encapsulation efficiency as compared to microcapsules loaded with rosemary essential oil. Interestingly, cinnamon bark oil was found more effective as it offered better functional properties compared to rosemary essential oil.

In the third part of the research, an attempt was made to prepare microcapsules using pullulan as a shell material and thyme oil as a core material. It was interesting to see higher encapsulation efficiency (>85%) of pullulan microcapsules compared to microcapsules

prepared in the other parts of the research. The oil release kinetics of the microcapsules was found to be governed by diffusion mechanism. The result revealed retention of more than 16% of the initial oil present on the finished fabric even after 20 washes. Hence, the better mosquito repellency of the finished fabric was observed even after 20 washes compared to the finished fabrics developed in previous parts of the research. In this research, the finished fabric exhibited 95.21% and 93.11% antibacterial activity against *S. aureus* and *E. coli* bacteria, respectively. The finished fabric also showed more than 90% antioxidant activity and a pleasant aroma. The exciting trends in the durability of functional properties were obtained. The result revealed that mosquito repellency was subject to the presence of aroma on the finished fabric. Hence, the mosquito repellency was retained till the fabric remained fragrant. However, the antibacterial and antioxidant activity was subject to the amount of phenolic content on the finished fabric. Once the amount of phenolic content gets reduced, the effectiveness of the finish toward bacterial colony reduction and radical scavenging was also reduced.

सार

21वीं सदी में घातक वायरस, मच्छर जनित और रोगजनक रोगों की घटनाओं में काफी वृद्धि हुई है। इन बीमारियों के प्रसार से बचने के लिए सबसे अच्छी रणनीतियों में से एक सुरक्षात्मक कपड़ा उत्पादों जैसे कार्यात्मक परिधान (जीवाणुरोधी, ऑक्सीकरणरोधी, विषाणुरोधी, मच्छर प्रतिरोधी), कार्यात्मक मुखौटा, दुपट्टा, लहंगा, पर्दे और चादर का उपयोग करना है। अत्यधिक जहरीले और कृत्रिम रसायनों के विकल्प खोजकर वस्त्रों के कार्यात्मककरण के लिए नई रणनीतियों का पता लगाया जा रहा है। प्राचीन काल से औषधीय प्रस्तावों के लिए तात्विक तेल का उपयोग उनके अंतर्निहित जैविक गुणों के कारण किया जा रहा है। हालांकि, वस्त्रों के कार्यात्मककरण के लिए तात्विक तेल का उपयोग उनकी अस्थिर प्रकृति के कारण सीमित है। वस्त्रों के माध्यम से उत्कृष्ट औषधीय प्रभाव प्राप्त करने के लिए उनके स्थायित्व को बढ़ाने के लिए कई प्रयास किए गए हैं। वर्तमान शोध कार्यात्मक बहुलक में माइक्रोकैप्सुलेटेड नया तात्विक तेल का उपयोग करके बहुक्रियाशील वस्त्रों के विकास के इर्द-गिर्द घूमता है।

इस शोध के पहले भाग में अजवायन तेल से भरी हुई इन-सीटू संश्लेषित काइटोसान-फॉस्फेट माइक्रोकैप्सूल विकसित करने का प्रयास किया गया था। माइक्रोकैप्सूल की एनकैप्सुलेशन उपज और एनकैप्सुलेशन दक्षता को मापकर बहुलक-से-तेल एकाग्रता को अनुकूलित करने के लिए सत्रह प्रयोग किए गए। प्राप्त माइक्रोकैप्सूल ने हर प्रयोग में 71% से अधिक एनकैप्सुलेशन दक्षता की पेशकश की जो काफी महत्वपूर्ण था। विकसित माइक्रोकैप्सूल कार्यात्मक गुणों को प्रदान करने के लिए एक ऐक्रेलिक बंधक का उपयोग करके लिनन के कपड़े पर लगाए थे। माइक्रोकैप्सूल गोलाकार थे और एक आव्यूह प्रकार की आकृति विज्ञान के स्वामित्व में थे। दिलचस्प बात यह है कि माइक्रोकैप्सूल से निकलने वाला तेल प्रसार और सूजन तंत्र द्वारा नियंत्रित होता है। परिणाम प्रभावशाली थे, जिसने 20 बार धोने के बाद भी कपड़े पर तेल की उपस्थिति को दिखाया। तात्विक तेल अत्यधिक ज्वलनशील होते हैं; हालांकि, इस शोध ने अजवायन के तेल से भरी अग्नि मंदता माइक्रोकैप्सूल तैयार करने का प्रयास किया। तैयार कपड़े ने उत्कृष्ट मच्छर भगाने (100%), ऑक्सीकरणरोधी गतिविधि (>96%), सुखद सुगंध और मध्यम अग्नि मंदता का प्रदर्शन किया। तैयार कपड़े ने बार-बार धोने के बाद भी उत्कृष्ट जीवाणुरोधी गतिविधि का प्रदर्शन किया। कपड़े के यांत्रिक गुणों और वायु पारगम्यता में मामूली परिवर्तन देखा गया। तात्विक तेल (गुलमेहंदी तात्विक तेल) को बदलकर इस शोध की प्रामाणिकता की पुष्टि करने का एक और प्रयास किया गया था। इसके अलावा, परिणाम पिछले

अनुभाग में देखे गए समान रुझान दिखाते हैं। ये परिणाम हमें माइक्रोकैप्सूल की एनकैप्सुलेशन दक्षता बढ़ाने और बेहतर कार्यात्मक गुण प्राप्त करने के लिए नए बहुलक और तेल का पता लगाने के लिए प्रेरित करते हैं।

शोध के दूसरे भाग में माइक्रोकैप्सूल के एनकैप्सुलेशन दक्षता और माइक्रोकैप्सूल से निकलने वाले तेल की गतिकी पर बहुलक मिश्रण के प्रभाव का पता लगाने का प्रयास किया गया था। माइक्रोकैप्सूल को काइटोसान-जिलेटिन के मिश्रण का उपयोग खोल सामग्री के रूप में और गुलमेहंदी के तेल को मुख्य सामग्री के रूप में उपयोग करके विकसित किया गया था। तैयार किए गए माइक्रोकैप्सूल ने आव्यूह और कुण्ड प्रकार की आकृति विज्ञान को दिखाया। माइक्रोकैप्सूल ने एक निरंतर तेल निकलने के रेखा-चित्र की पेशकश की, जो एक प्रसार तंत्र द्वारा शासित थी। प्राप्त माइक्रोकैप्सूल ने 68% से अधिक एनकैप्सुलेशन दक्षता की पेशकश की। तैयार कपड़े ने उत्कृष्ट मच्छर भगाने, जीवाणुरोधी गतिविधि, ऑक्सीकरणरोधी गतिविधि और बार-बार धोने के बाद भी एक सुखद सुगंध दिखाई। तात्विक तेल (दालचीनी की छाल का तेल) को बदलकर शोध की प्रामाणिकता की पुष्टि करने का एक और प्रयास किया गया था। प्राप्त माइक्रोकैप्सूल ने एक समान प्रकार की आकृति विज्ञान और तेल के निकलने की तंत्र की पेशकश की जैसा कि शोध के दूसरे भाग के पिछले खंड में पाया गया था। हालांकि, गुलमेहंदी तात्विक तेल से भरे हुए माइक्रोकैप्सूल की तुलना में माइक्रोकैप्सूल ने बेहतर एनकैप्सुलेशन दक्षता की पेशकश की। दिलचस्प बात यह है कि दालचीनी की छाल का तेल अधिक प्रभावी पाया गया क्योंकि यह गुलमेहंदी तात्विक तेल की तुलना में बेहतर कार्यात्मक गुण प्रदान करता है।

शोध के तीसरे भाग में पुलुलन को खोल सामग्री के रूप में और अजवायन तेल को मुख्य सामग्री के रूप में उपयोग करके माइक्रोकैप्सूल तैयार करने का प्रयास किया गया था। शोध के अन्य भागों में तैयार किए गए माइक्रोकैप्सूल की तुलना में पुलुलन माइक्रोकैप्सूल की उच्च एनकैप्सुलेशन दक्षता (>85%) देखना दिलचस्प था। माइक्रोकैप्सूल के तेल निकलने की गतिकी को प्रसार तंत्र द्वारा नियंत्रित किया गया था। परिणाम में 20 बार धोने के बाद भी तैयार कपड़े पर मौजूद प्रारंभिक तेल के 16% से अधिक की अवधारण का पता चला। इसलिए, शोध के पिछले भागों में विकसित किए गए तैयार कपड़ों की तुलना में तैयार कपड़े की बेहतर मच्छर भगाने की क्षमता 20 बार धोने के बाद भी देखी गई। इस शोध में तैयार कपड़े ने क्रमशः एस. ऑरियस और ई. कोलाई जीवाणु के खिलाफ 95.21% और 93.11% जीवाणुरोधी गतिविधि का प्रदर्शन किया। तैयार कपड़े ने 90% से अधिक ऑक्सीकरणरोधी गतिविधि और एक सुखद

सुगंध भी दिखाया। कार्यात्मक गुणों के स्थायित्व में रोमांचक रुझान प्राप्त किए गए थे। परिणाम से पता चला कि मच्छर भगाने की क्रिया तैयार कपड़े पर सुगंध की उपस्थिति के अधीन थी। इसलिए, कपड़े के सुगंधित रहने तक मच्छर भगाने की क्षमता बरकरार रखी गई। हालांकि, जीवाणुरोधी और ऑक्सीकरणरोधी गतिविधि तैयार कपड़े पर फेनोलिक सामग्री की मात्रा के अधीन थी। एक बार जब फेनोलिक सामग्री की मात्रा कम हो जाती है, तो जीवाणु आबादी में कमी और ऑक्सीकरणरोधी गतिविधि की प्रभावशीलता भी कम हो जाती है।

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