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The Functionalization of Cotton Fabric Using *Urtica dioica* Microcapsules to Develop UV Protective Textiles

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ABSTRACT
The World Health Organization (WHO) and the United Nations Environment Programme (UNEP) have highlighted the increased risk of skin cancer due to exposure to UV radiation. This emphasizes the importance of safeguarding the skin. Although sunscreens are commonly used for protection their high cost, the need for frequent reapplication, and pollution in coastal areas necessitate alternative solutions. Clothing also offers protection but requires additional application of dyes and finishes for prolonged effectiveness. The application of synthetic or natural dyes can enhance the UV protection properties of the fabric but Synthetic dyes contribute to pollution, while natural dyes lack durability. Addressing these challenges, this study focuses on providing a durable and sustainable solution for enhancing the UV protective properties of cotton fabric using Urtica dioica microcapsules based on RSM design. Urtica dioica plant extract serves as the core material for the microcapsules. Design Expert software 10 has been used for the study. It was observed through software that at 4.116% concentration of microcapsules, 3% concentration of binder and a curing temperature of 113.258 °C an optimum UPF with sufficient fabric properties can be achieved. On conducting the experiments on these optimum conditions UPF, tensile strength and bending length value were found to be 277.60, 14.89 N/cm^2 and 2.00 cm respectively. Results indicate that cotton fabric demonstrates excellent UV protection properties even after the 20th wash cycle. This research contributes to the ongoing quest for fabric with UV protection, emphasizing both efficacy and sustainability compared to current synthetic alternatives available in the market.

KEYWORDS
cotton fabric, Urtica dioica, microencapsulation, UV protection

INTRODUCTION

The sun plays a crucial role in sustaining life on Earth, providing essential light and energy. However, the ultraviolet (UV) radiation emitted by the sun, while beneficial in many ways, poses significant risks to human health. Exposure to UV radiation has been linked to various health issues such as skin cancers and eye cataracts [1,2].

According to estimates by the UNEP millions of cases of skin cancer can be found in the USA by the year 2075 because of UV radiation [3].

Recognizing the importance of protecting ourselves from the harmful effects of the sun, people have sought various measures. Sunscreen is a commonly used protective agent, but it comes with limitations. Sunscreen needs to be reapplied, works only in limited areas, and can contribute to water pollution near coastlines [4,5]. Moreover, concerns have been raised about the accumulation of chemical sunscreen in the body, leading to potential hormonal imbalances, cancer, and allergic reactions [6].

Clothing also serves as a protective barrier against UV rays, but not all fabrics offer sufficient protection. Some synthetic UV absorbers are available in the market, but they come with health hazards. Nanoparticles of Zinc and Titanium have been explored to enhance the anti-UV properties of fabrics [7-9]. However, research suggests that these particles may have adverse effects on both the environment and human health [10]. The scientific committee of the EU has also reported the high risk of diseases caused by nanoparticles [11].

In addition to these concerns, the high cost of nanoparticles poses a drawback to their widespread use. Natural dyes and finishes have been employed to enhance the UV protective properties of fabrics, but their durability remains a significant concern for researchers [12-18]. The UV protection of dyed fabrics may decrease due to solarization from exposure to sunlight and colour fading during the laundry process [19].

Given these limitations and concerns, this research aims to optimize the application of *Urtica dioica* (*Nettle*) microcapsules on Cotton fabric for UV protection while maintaining other important properties. Several plant resources such as *Urtica dioica, Trapa natans, Musa acuminata, Girardinia diversifolia, Malus domestica, Murraya koenigii, Psidium guajava* were collected for screening purposes*.* *Urtica dioica* was selected based on its UPF properties and abundance availability. *Urtica dioica* plant, often considered a weed, is commonly found in moist, shady, and anthropogenic environments. The genus consists of 46 species, notably *Urtica dioica* (stinging nettle) and *Urtica urens* (small nettle), native to Europe, Africa, Asia, and North America, with naturalized populations in other temperate regions worldwide [20]. It has also been reported by Durovic et al. that *Urtica dioica* leaves showed the highest concentration of Polyphenolic compounds especially *hydroxycinnamic* compounds [21]. Cotton is a preferred fabric during the peak of the UV Index in summer, and *Urtica dioica* is abundantly available as a weed plant in the Himalayan region of India [22]. Physical properties such as tensile strength and bending length are crucial considerations in the application of a plant extract finish.

To achieve the objectives of this study, process parameters were optimized using the Box and Behnken design under response surface methodology (RSM) using Design Expert Software Version 10. It's worth noting that the focus of this paper is on the application aspect of microcapsules rather than their formulation.

In conclusion, the research seeks to address the limitations of current UV protection measures by exploring a sustainable and effective solution through the application of *Urtica dioica* microcapsules on Cotton fabric. By doing so, the study aims to contribute to the development of safer and more environmentally friendly alternatives for UV protection in textiles.

EXPERIMENTAL

Materials and Methods

The study employed a Soxhlet Apparatus for methanol (80%) extraction of *Urtica dioica* leaf powder, followed by phytochemical analysis based on Brain and Turner's guidelines [23]. Ten grams of powder of each selected plant material was individually extracted through the Soxhlet apparatus with 80 per cent of the methanol for 24 hours at 40 °C. Extracts were filtered using filter paper and the filtrate was used for phytochemical screening and the estimation of total phenolic content. Microcapsules, formulated using sodium alginate(Loba Chemie Pvt LTD, India) and carrageenan(Seaweed fertiliser, Gujrat), were applied to cotton fabric via the exhaust cum pad-dry-cure method with an acrylic binder (Sakshi Dyes and chemicals, Delhi).

Process optimization utilized the Box and Behnken design, considering microcapsule concentration, binder concentration, and curing temperature. The experimental matrix, analyzed using Response Surface Methodology (RSM) in Design Expert software, featured 17 samples. The complete preparation process has been subjected to a patent process. This paper deals with the application part only. The least square method and multiple regression analysis established a mathematical relationship between microcapsule parameters and fabric characteristics (UPF, tensile strength, bending length), expressed through a polynomial equation.

Fabric physical attributes, including tensile strength and bending length, were assessed alongside UV protection properties using Innolab's tensile strength tester and standard methods IS 1969–1, 2009, and ASTM D1388-96, 2002. Standard atmosphere conditions for all testing the samples were 20 °C (68 °F) and 65% relative humidity. The washing durability test, employing AATCC method 61 (1996) test no. 2A on a Metrex launder-O-Meter, examined the longevity of the UV-protective finish after 20 washes, with UPF evaluations after five, ten, fifteen, and twenty washes. Scanning Electron Microscopy (SEM) analysis at G. B. Pant University utilized a JEOL JSM-6610 LV SEM to verify the presence of microcapsules in treated fabric samples, featuring images captured at various magnifications. Standard test method AATCC-183: 2004 was used for calculating UPF (23 AATCC 183:2004. 2010). The rating has been given according to the UPF Classification system (AS/NZS 4399:1996) [24-27].

Table 1. UPF Classification system (AS/NZS 4399:1996)

UPF Rating	UVR Protection	Effective UVR transmission (%)	UPF labelling
15-24	Good protection	6.7-4.2	15, 20
25-39	Very good protection	4.1-2.6	25,30,35
40-50,50+	Excellent protection	≤ 2.5	40,45,50,50

RESULTS AND DISCUSSION

The study examined a plain weave white Khadi cotton fabric, revealing key physical properties. It is clear from Table 2 that the fabric had a count of 26 warp x 20 weft per cm a weight of 0.970 GSM, and a thickness of 0.28 mm. The breaking strength was measured at 19 N/cm^2, and the bending length indicated flexibility (1.4 cm). The UPF value of the fabric was observed to be 9.9 which means the fabric is not able to provide UV protection. The provided fabric properties offer valuable insights into the characteristics of the plain weave khadi cotton fabric. The warp and weft count of 65 and 50 per inch, respectively, indicate a moderately dense weave, influencing the fabric's appearance and durability. With a weight of 0.970 grams per square meter (GSM) and a thickness of 0.28 mm, the fabric exhibits a balance between substance and lightness, suggesting versatility in potential applications. The breaking strength of 19 N/cm^2 signifies its ability to withstand tensile forces, while the bending length of 1.4 cm suggests a moderate level of flexibility, crucial for comfort and ease of movement.

Table 2. Specifications of khadi cotton fabric

S. No.	Fabric Properties (units)	Observed values
1.	Fabric count	65(warp) * 50(weft)
2.	Fabric weight(GSM	0.97
3.	Fabric thickness(mm)	0.28
4.	Fabric strength(N/cm^2)	19.00
5.	Fabric bending length(cm)	1.90
6.	UPF	9.9

Phytochemical Analysis of the *Urtica dioica* leaves extract revealed the presence of alkaloids, flavonoids, phenols, tannins, and anthraquinonoids, with tannins and flavonoids identified as prominent constituents. Due to their complex structure, these compounds effectively function as UV absorbers. To enhance durability, the microencapsulation method was chosen. The application of *Urtica dioica* microcapsules on cotton fabric was optimized using a Box and Behnken design in the current investigation, employing Response Surface Methodology (RSM) for analysis. The outcomes of the experiments, along with the design matrix, are presented in Table 3, detailing the parameters for the successful application of *Urtica dioica* microcapsules on cotton fabric.

Table 3. Finishing conditions with observed responses for application of *Urtica dioica* microcapsules on cotton fabric

		Factor 1	Factor 2	Factor 3	R1	R2	R3
Std	Run	A: Concentration of microcapsules %	B: Concentration of Binder %	C: Curing temperature °C	UPF	Tensile strength N	Bending length cm
3	1	1	2	140	30.2	16.02	1.7
11	2	1	1	120	26	18.26	1.6
6	3	1	3	120	36.8	17.02	1.8
7	4	1	2	100	32.2	17.96	1.5
14	5	3	3	100	103.4	16.98	1.9
10	6	3	1	100	64.7	18.76	1.4
1	7	3	2	120	69.2	17.26	1.5
8	8	3	1	140	41.7	18.98	1.7
5	9	3	2	120	71.2	17.25	1.5
13	10	3	2	120	71.2	17	1.5
16	11	3	2	120	71.2	17.03	1.5
2	12	3	3	140	221.3	16.87	2.1
9	13	3	2	120	133.4	17.2	2
12	14	5	2	100	156.6	17.88	2.2
4	15	5	1	120	184.5	18.56	2.3
17	16	5	2	140	235.8	16.23	2.4
15	17	5	3	120	389.3	16.23	2.5

Note: Std. (Run as given by Design Expert Software version 10); R1: Response -1 (UPF); R2: Response 2 (Tensile Strength); R3 :Response -3 (Bending Length)

Microcapsules failed to form under various combinations of independent variables in the extrusion method. The optimal conditions for microcapsule formation were identified as a 1% concentration of the wall material with an M:L ratio of 1:50. Additionally, a 1% concentration of calcium chloride was determined as the optimal condition for microcapsule formation. The statement "microcapsules failed to form" suggests that the intended process of encapsulating core materials within a shell was unsuccessful. This could be due to various factors, such as inadequate chemical concentrations or incorrect ratios, unfavourable physical conditions like temperature or pH, chemical incompatibility between materials, or the occurrence of aggregation instead of individual microcapsule formation. Optimizing these factors is essential for achieving successful microencapsulation.

Figure 1 provides a microscopic image of *Urtica dioica* extracts' microcapsules at 100x magnification.

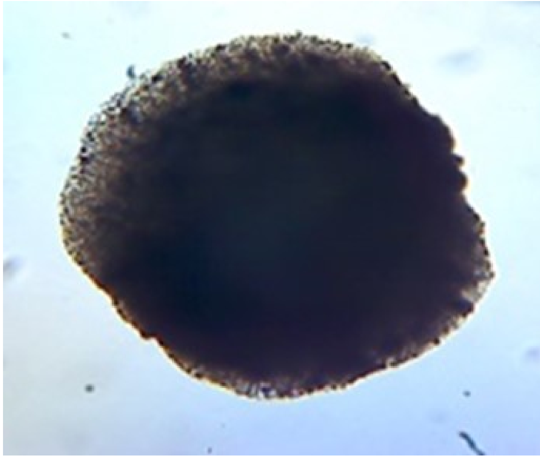


Figure 1. *Urtica dioica* Microcapsules at 100X magnification

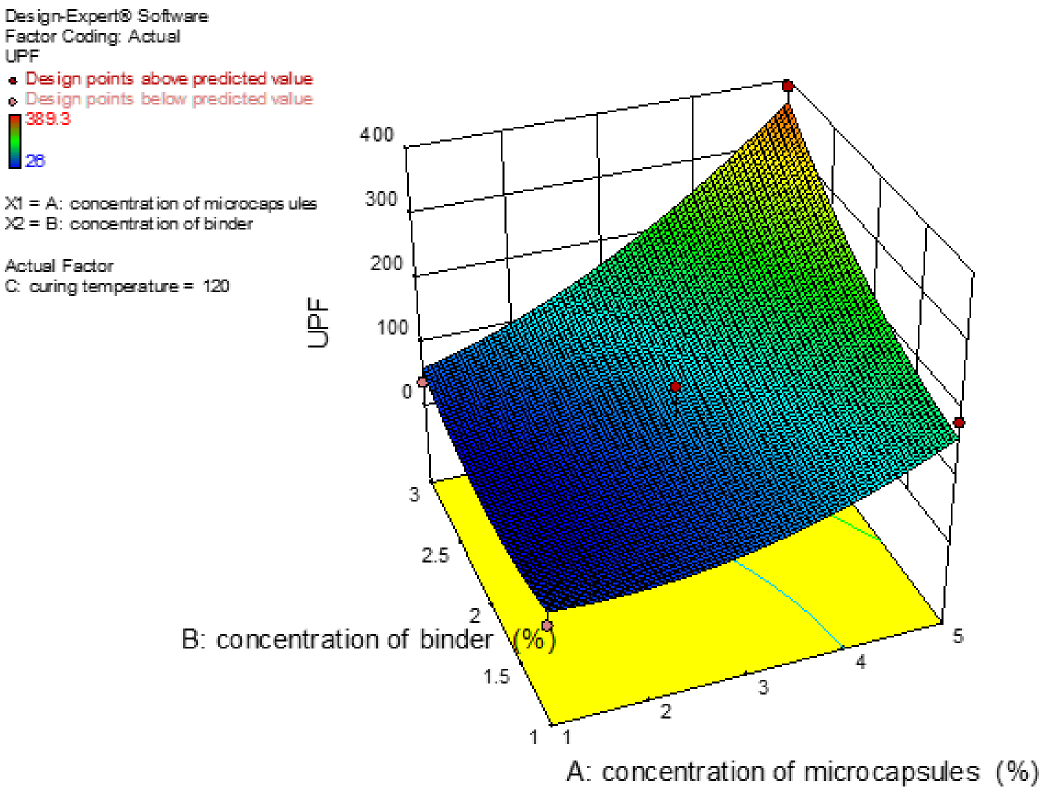
Effect of Parameters on UPF

The study investigated the impact of independent variables on responses (dependent variables) in the application of *Urtica dioica* microcapsules on cotton fabric. Regression analysis, considering the UPF as the response, yielded a significant model as indicated by the F-value. The least square regression analysis resulted in a quadratic polynomial equation describing the relationship between UPF (Y1) and finishing variables (A, B, C). Model reduction retained significant terms, yielding a final equation that demonstrated the influence of the concentration of microcapsules (A), the concentration of binder (B), and their interaction (AB) on UPF. The adequacy of the model was testified by coefficients of determination (R^2). The value of determination coefficients (R^2) of UPF was 0.9521, which implied that the 95.21% variation in the results of UPF was explained by the quadratic polynomial model. The value of adequate precision was 14.538 which was greater than 4 (signal-to-noise ratio). Therefore model was adequate to predict the response and interpret the effect of variables on the UPF.

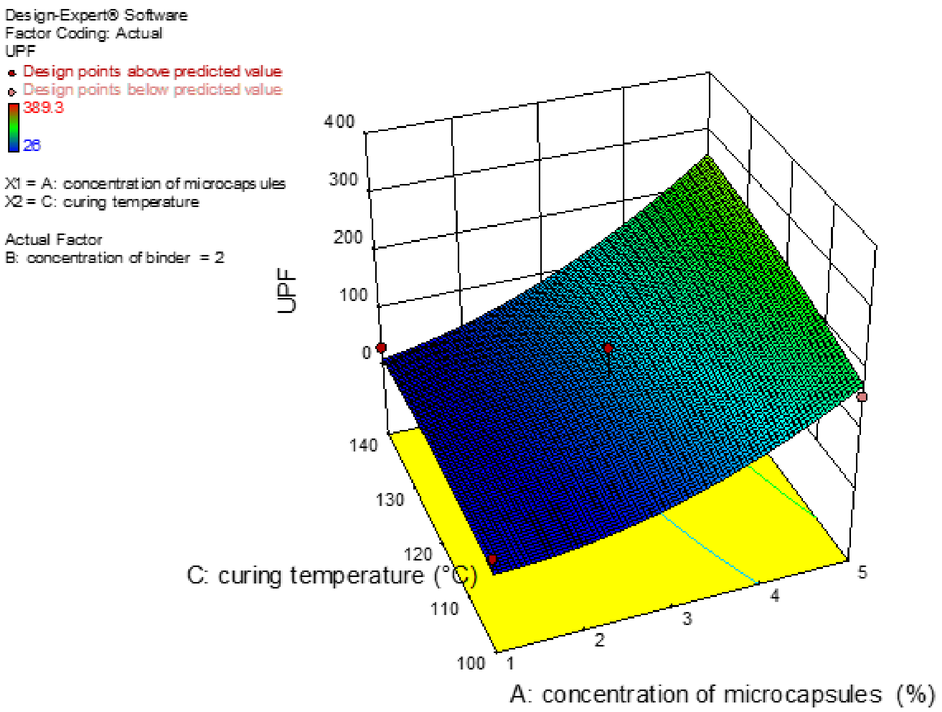
$$UPF\ (Y1) = 83.24 + 105.125\ A + 54.2375\ B + 48.5\ AB + 40.9175\ A^2 \tag{1}$$

Response surface plots illustrated the interactive effects, revealing increased UPF values with higher concentrations of microcapsules and binder. The study concluded that the concentration of microcapsules, concentration of binder, and curing temperature were key predictors for UPF, with their combined effects enhancing UV protection in the finished cotton fabric. Three-dimensional surface plots visually depicted these relationships.

Figure 2(a) shows the interactive effect of concentration of microcapsules (A) and concentration of binder (B) on UPF. It is clear from the figure that the highest UPF value was obtained at the interaction of a higher level of concentration of microcapsules with a higher level of concentration of binder. It is evident from Figure 2(b) that the effect of curing temperature on UPF was negligible while the concentration of microcapsules positively affected the UPF values of the finished fabric. This implied that as the concentration of microcapsules was increased, an increase was observed in UPF values. Figure 2(c) shows the interactive effect of the concentration of binder (B) and curing temperature (°C). At higher curing temperatures, the concentration of the binder positively affects the UPF of the finished fabric. A higher UPF value was observed at the interaction of the maximum level of concentration of binder with curing temperature. It has also been reported by Abdelkhalik et al. a natural component of Turmeric with melamine polyphosphate (MPP) also enhanced the UPF of the fabric when applied to cotton fabric (28).



a)



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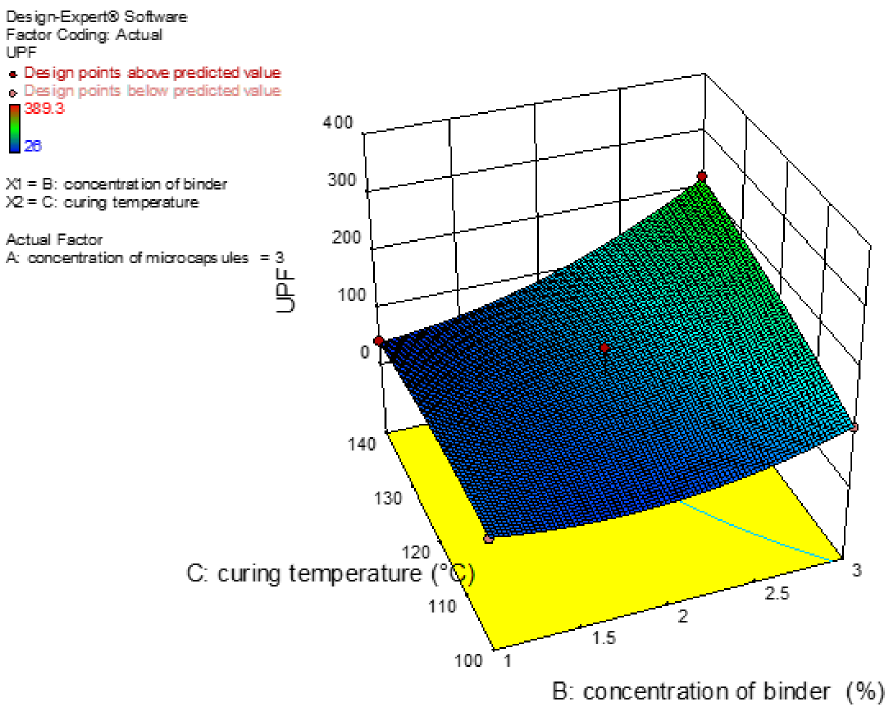
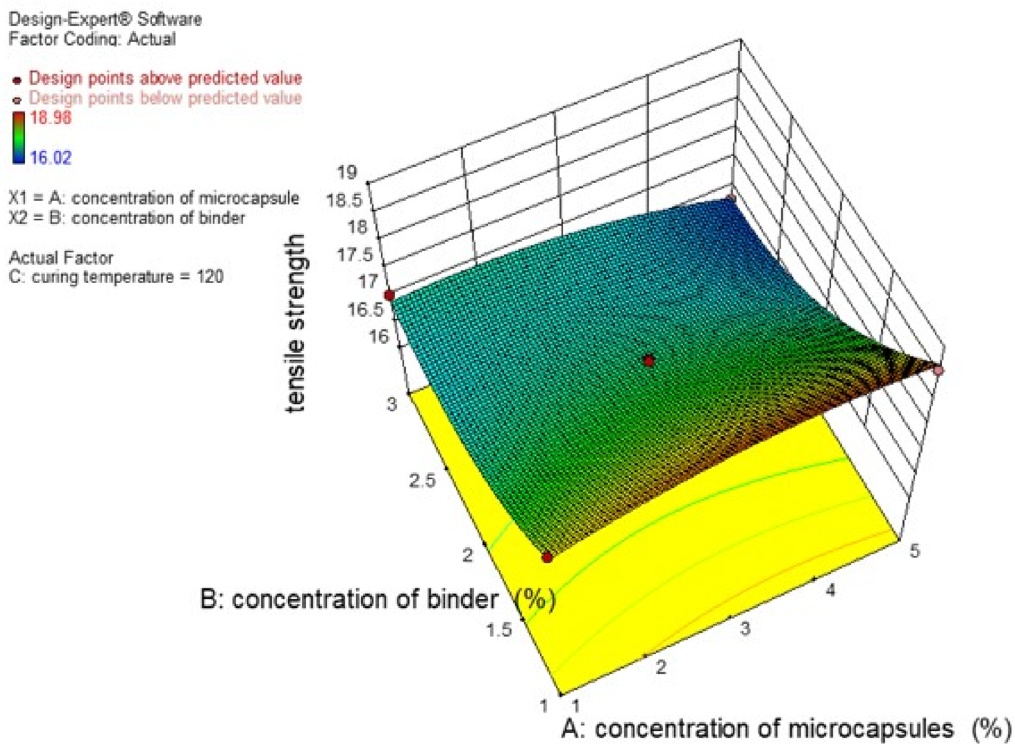


c)

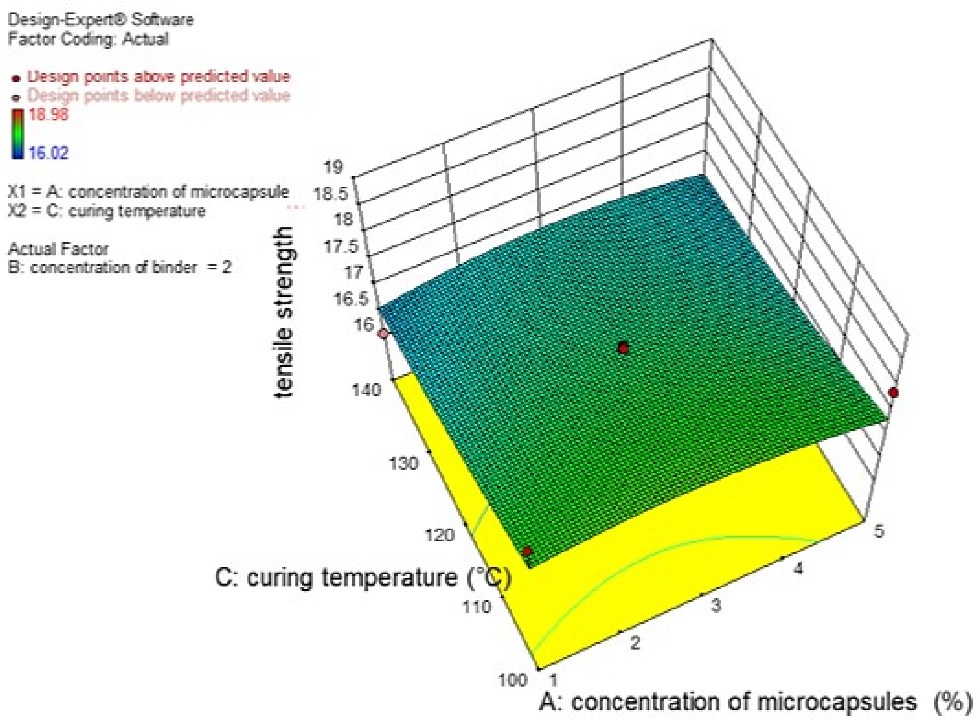
Figure 2. The combined effect of a) concentration of binder and microcapsules on UPF; b) curing temperature and concentration of microcapsule on UPF; c) curing temperature and concentration of binder with UPF

Effect of parameters on tensile strength

It is clear from Fig 3 a), 3 b) and 3 c) indicating an increase in the concentration of microcapsules, concentration of binder, and curing temperature leading to increased tensile strength. The combined effect of concentration of microcapsules and concentration of binder, as well as concentration of binder and curing temperature, decreased tensile strength, while the combination of concentration of microcapsules and curing temperature had a positive effect. Quadratic terms indicated minimum tensile strength at the centre point of concentration of microcapsules and maximum at the centre point of concentration of binder and curing temperature. Three-dimensional surface plots were developed for tensile strength to visualize the effects between two independent variables while keeping the third variable at an optimum point.



a)



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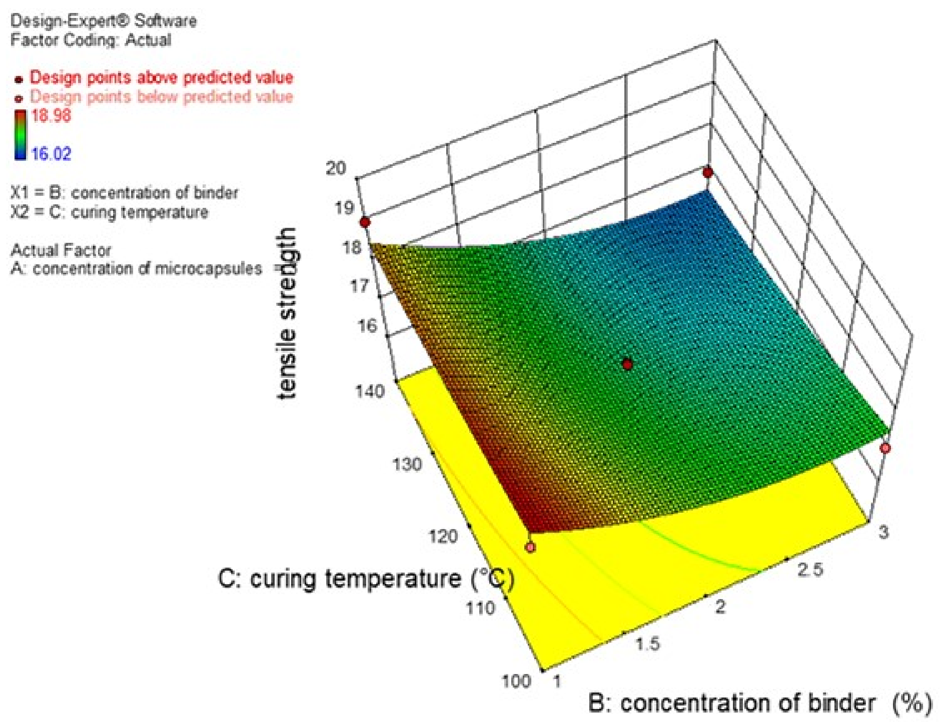
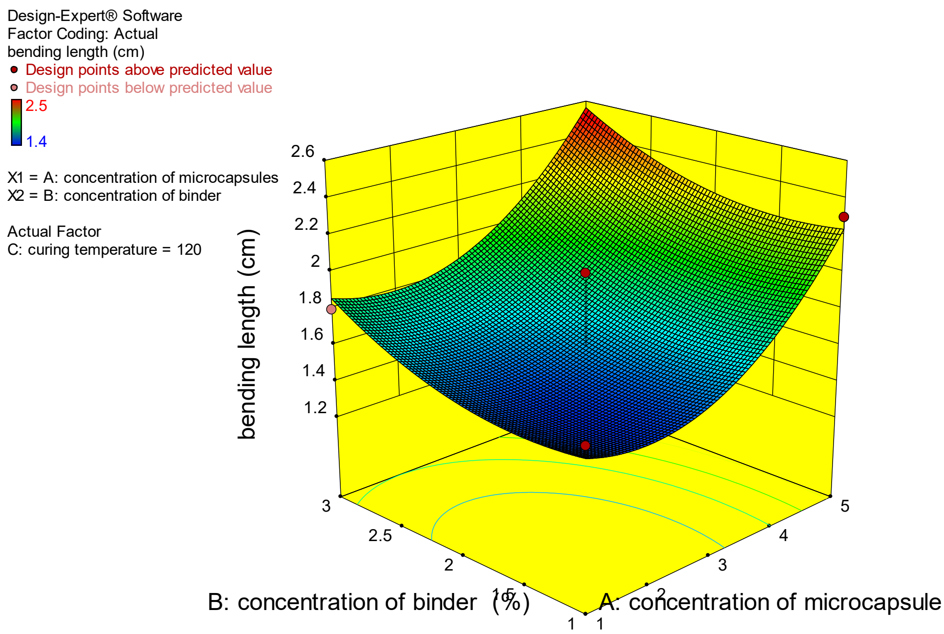


c)

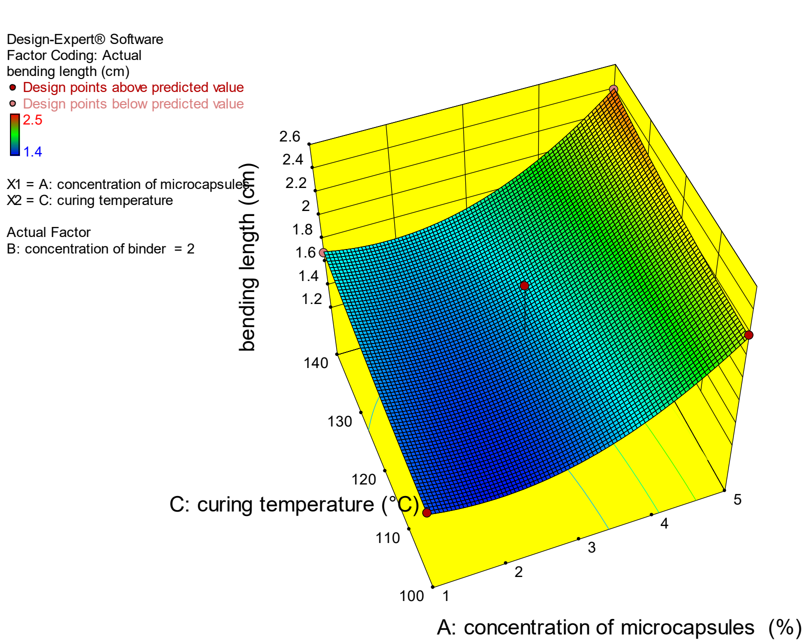
Figure 3. The combined effect of a) concentration of binder and microcapsules on Tensile strength; (b) curing temperature and concentration of microcapsules on tensile strength; c) curing temperature and concentration of binder on tensile strength

Effect of parameter on Bending length

It is clear from Figures 4 (a), 4 (b), and 4 (c) that the combined effect of the concentration of microcapsules and concentration of binder, the concentration of microcapsules and curing temperature, as well as the concentration of binder and curing temperature, increased the bending length. Quadratic terms suggested that the maximum bending length occurred at the centre point of concentration of microcapsules and concentration of binder, while it was minimum at the centre point of curing temperature. Three-dimensional surface plots were developed for bending length to visualize the effects between two independent variables while keeping the third variable at an optimum point.



a)



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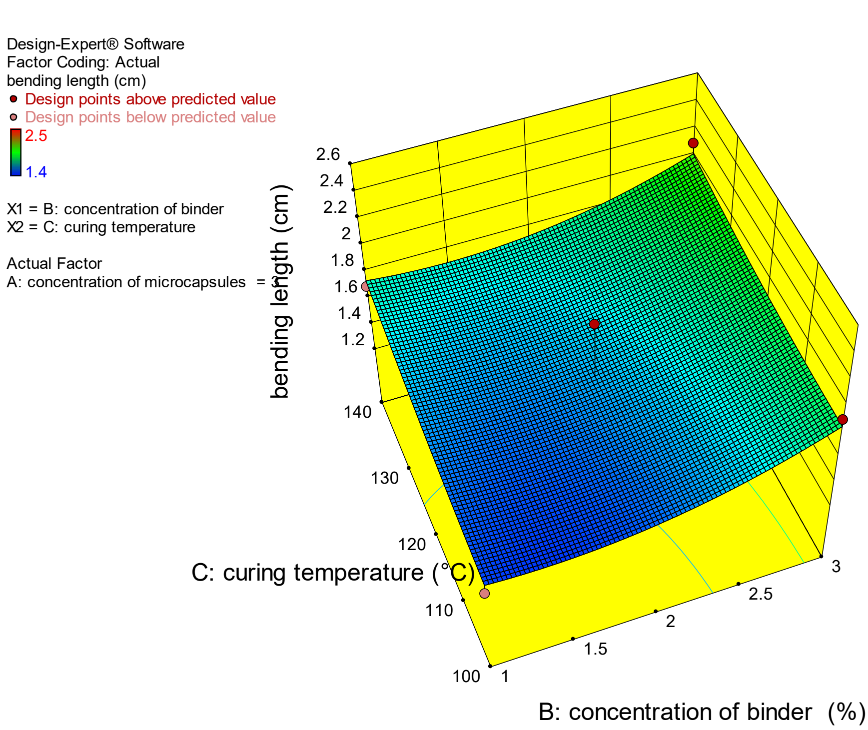


c)

Figure 4. The combined effect of a) concentration of microcapsules and binder on bending length; b) concentration and curing temperature on bending length; c) curing temperature and concentration of binder on bending

Figure 4 (a) depicts the interactive effect of concentration of microcapsules (A) and concentration of binder (B) on bending length, revealing the highest bending length at increased concentration of microcapsules and high concentration of binder. Conversely, the minimum bending length was observed at the lower concentration of binder with a lower level of concentration of *Urtica dioica* microcapsules. Figure 4 b) shows the interactive effect of the concentration of microcapsules (A) and curing temperature (C) on bending length, with the maximum bending length observed at the highest concentration and maximum curing temperature. Figure 4 c) illustrates the interactive effect of concentration of binder (B) and curing temperature (C) on bending length, indicating the maximum bending length at high curing temperature with a high concentration of binder. The regression model for predicting bending length concludes that the best predictors are the concentration of microcapsules and binder at the linear level, the interaction of both with each other and the concentration of microcapsules at the quadratic level.

Optimized finishing conditions for application of *Urtica dioica* microcapsules on cotton fabric

The best results of finishing were achieved with 4.11%, 3% and 113.25 °C for the concentration of microcapsules, concentration of binder and curing temperature respectively. Further validation experiment results indicate that the experimental results were similar to the predicted theoretical values (Table 4).

It can be concluded from the results that the model was sufficient. Therefore, the optimized value of the concentration of *Urtica dioica* microcapsules (A), the concentration of binder (B) and curing temperature (C) predicted by the numerical optimization i.e. 4.11% concentration of microcapsules with 3% concentration of binder at curing temperature of 113.25 °C were selected as optimum conditions for UV protective finishing of cotton fabric with *Urtica dioica* microcapsules.

To determine the validity of the optimized conditions, a set of experiments was conducted according to the conditions that were optimized by the model. The experiments were run in triplicates and the average value was calculated. The predicted results obtained from the equation for the application of *Urtica dioica* plant microcapsules on cotton fabric using the optimized conditions are shown in Table 3. The average value of the experiment was compared with the predicted values obtained. The results indicate that the experimental results were quite close to the predicted theoretical values.

Table 4. Predicted and experimental values of *Urtica dioica* microcapsules finished cotton fabric with optimum finishing conditions

S.NO.	Response	Predicted value	Experimental value
1.	UPF	246.94	277.60
2.	Tensile strength(N/cm^2)	16.75	14.89
3.	Bending length(cm)	2.16	2.00

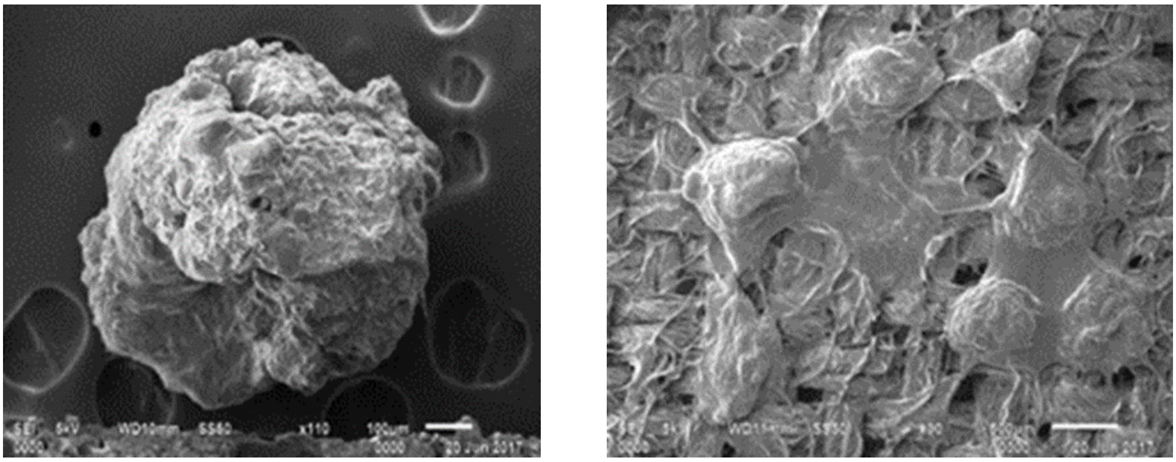
It can be concluded from the results that the model was sufficient.

SEM analysis

Figure 4 provides an SEM analysis of microcapsules and their attachment with the binder on the surface of cotton fabric. The microcapsules maintain their spherical shape, showcasing a successful bonding with the fabric.

SEM analysis is a powerful tool that allows for a detailed examination of the surface morphology of materials. In the context of this study, it provides valuable insights into the interaction between the microcapsules and the cotton fabric. The spherical shape retention is noteworthy, indicating the effectiveness of the bonding process and the stability of the microcapsules on the fabric surface.

This observation is significant in validating the proposed application of *Urtica dioica* microcapsules on Cotton fabric for UV protection. The SEM analysis serves as a visual confirmation of the successful integration of the microcapsules, further supporting the potential of this approach in enhancing the UV protective properties of the fabric.



a) b)

Figure 5. SEM of a) microcapsules of *Urtica dioica*; b. microcapsules of *Urtica dioica* on surface of cotton fabric

Wash Durability:

Table 5 represents the results of washing durability of fabric samples finished with *Urtica dioica* microcapsules. This table shows that excellent UV protection properties were observed to remain in the case of each finished fabric sample even after 20 wash cycles. It was observed from Table 4 that in the first 5 cycles, the UPF started to increase from 277.6 to 312.4. This may be due to the shrinkage of cotton fabric. Further from the 5^{th} to 20^{th} washes, It starts to deteriorate. The reason for this deterioration may be due to the removal of microcapsules from the surface.

Table 5. Durability of microcapsules

No. of a wash cycle	UV protection property	
	UPF	Category
0	277.6	Excellent
5	312.4	Excellent
10	271.7	Excellent
15	221.1	Excellent
20	206.8	Excellent

CONCLUSION

The study concludes that the application of *Urtica dioica* microcapsules imparts UV protection properties to cotton fabric, demonstrating sustained efficacy even after 20 wash cycles. The ideal conditions for applying these microcapsules were identified, with an optimized concentration of 4.11% for microcapsules, 3.00% for binder, and a curing temperature of 113.25 °C. These parameters were determined as optimal for achieving UV-protective finishing on cotton fabric. The utilization of biodegradable plant waste, specifically *Urtica dioica* microcapsules, not only enhances UV protection with durability but also aligns with environmental sustainability.

Author Contributions

Conceptualization – Pargai D; methodology – Pargai D; formal analysis – Pargai D; investigation – Pargai D; resources – Pargai D; writing-original draft preparation – Pargai D; writing-review and editing – Pargai D and Jahan S; visualization – Pargai D; supervision Jahan S. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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