

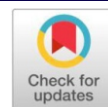
Original Research Article

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Textiles Enhanced with Nanoparticle-Infused Functional Finishes

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ABSTRACT

In light of the pervasive health threats posed by disease-carrying mosquitoes, such as those transmitting malaria and dengue, there is a growing need for effective mosquito repellents. Traditional repellents, including plant extracts, oils, and smoke, are increasingly being replaced by safer and eco-friendly alternatives. This study explores the development of mosquito-repellent textiles by synthesizing TiO₂ nanoparticles from *Ocimum basilicum* (basil) plant extracts in both pre- and post-calcinated forms. These nanoparticles were applied to cotton fabric through screen printing at two different concentrations and characterized using particle size analysis, Fourier transform infrared spectroscopy, X-ray diffraction, and scanning electron microscopy. The biosynthesis of calcinated nanoparticles resulted in an average grain size of 8.9 nm. Average sizes of 60-93 nm for calcinated nanoparticles and 113-146 nm for pre-calcinated nanoparticles were found by SEM analysis. Mosquito-repellent efficacy, assessed using cage tests with copper sulphate as a mordant, showed promising results. The repelling efficacy of fabrics treated with 10% pre-calcinated nanoparticles ranged from 90 to 96%; however, efficacy decreased following post-treatment. Wash durability testing showed that fabrics containing 10% post-calcinated nanoparticles performed their best after five wash cycles and were 50% effective after ten wash cycles. Despite the promising outcomes, the study faced challenges related to the uniform dispersion and adherence of nanoparticles during application, as well as the retention of functional efficacy after repeated laundering. Nonetheless, this study contributes to the growing body of knowledge on green nanotechnology by demonstrating the potential of basil leaf extract in combination with TiO₂ nanoparticles for developing sustainable, eco-friendly mosquito-repellent textiles.

Keywords: Ecofriendly textile finishes, Insect-repellent clothing, Nano finishing, Plant-based nanotechnology, Functional finishes, Sustainable fabrics, Basil nanoparticles, Cage test, Mosquito repellents, green synthesis.

Introduction

In the dynamic realm of the textile industry, protective textiles and functional finishes stand out as the fastest growing sector globally [29]. Essential to this growth is the role of functional finishes, particularly those safeguarding wearers and incorporating eco-friendly practices [10&30]. The term 'eco-friendly' signifies goods and services that leave minimal or no harm to the environment, the spirit increasingly embraced by consumers worldwide [7&9]. In response to this trend, a noteworthy development in textile finishes involves harnessing the medicinal properties of plant extracts [14& 16]. In India, a consumer-led movement is urging manufacturers to adopt clean technologies for producing eco-friendly textiles [4&12]. Amidst these concerns, the quest for sustainable solutions gains relevance in the realm of mosquito repellency [15&2]. Traditional repellents, laden with synthetic chemicals, raise environmental apprehensions. In contrast, natural repellents derived from plant sources are gaining traction [3].

This article explores the convergence of eco-friendly plant-based repellents with nanotechnology. The focus is on developing mosquito-repellent printed textiles, utilizing the unique properties of nanoparticles [7&26]. Emphasizing the green synthesis of nanoparticles for its cost-effectiveness and eco-friendliness, the study delves into the durability of the mosquito repellent finish through laundering-a pivotal consideration for the practical application of such textiles [20].

Materials & Methods

Nanoparticles were produced via a green synthesis method using fresh leaves of *Ocimum basilicum*. In the first stage of the synthesis, fresh leaves were rinsed and boiled in distilled water at 60°C for one to two hours, using a material-to-liquor ratio of 1:5 and filtered using a filter paper [31]. Titanium dioxide (TiO₂) served as the precursor for the synthesis of nanoparticles [11]. The plant extract was gradually added to the TiO₂ solution and the whole solution underwent continuous stirring at 400 rpm for 4 hrs using a magnetic stirrer. The solution was left to settle overnight, followed by centrifugation to eliminate by-products [5&8]. After processing, the resulting wet cake/slurry was dried for 12 hours at 70°C in a hot air oven, producing powdered pre-calcinated nanoparticles. This powder was calcinated again for two hours at 200°C in a muffle furnace to produce calcinated nanoparticles [17].

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The crystalline structure analysis of the nanoparticles was conducted using a Bruker D8 X-ray diffractometer. Debye-Scherrer's formula was used to calculate the average crystalline size. The formula is as follows: D is the average grain size, K is the Debye-Scherrer's constant (0.94), λ is the radiation wavelength, β is the full-width half maximum of the peak, and θ is the Bragg's angle. The average particle size was measured using a HORIBA SZ-100 particle size analyzer. FTIR spectral analysis was carried out to determine which functional groups were present in the nanoparticles. Using a scanning electron microscope (SEM - Model: ZEISS EVO MA 50) at a magnification of $\times 500 - \times 50.00$ and following normal protocols, the size of the nanoparticles was determined.

Print paste preparation

For the experiment, a plain-weave cotton fabric with 200 thread counts and 122 GSM was utilized. For efficient natural dye fixing, the de-sized and scoured fabric was pre-treated by soaking it in a solution of myrobalan powder and drying it in the sun. Fresh leaves of sweet basil were weighed, rinsed, steeped overnight, and boiled to obtain an aqueous extract (dye) for further printing [22]. The gum essential for printing was prepared from dried seed powder of Cassia tora. Simultaneous copper sulfate was selected as the mordanting agent. A 4:3 ratio of dye to gum was used to create the print paste, and 0.8 g of copper sulphate (a mordant) was added for every 100 g of gum. Five distinct varieties of print pastes that deter mosquitoes were created by individually adding nanoparticles at concentrations of 5% and 10% in two distinct forms (pre- and post-calcinated) [1]. Using the screen-printing technique, the mosquito repellent paste was applied to the cotton fabric that had already been treated. The printed fabrics underwent a post-treatment that included steaming, steeping in a sodium chloride solution, and two days of shade drying. After that, they were rinsed with a 2 g/L neutral detergent solution. This was done to improve the fastness attributes of the materials. The fabrics were then subjected to shade drying. This post-treatment procedure facilitates the effective adhesion of nanoparticles to the fibers, the elimination of surplus dye and nanoparticles from the fabric's surface, enhancing its general fastness qualities [28]. To determine the proportion of efficacy lost, mosquito repellent tests were conducted on the fabrics both before and after post-treatment.

Assessment of Mosquito repellent efficacy

Before conducting tests, the fabric samples were conditioned by placing in a controlled environment for 24 hours at 65 ± 2 percent humidity and a temperature of $210^\circ\text{C} \pm 20^\circ\text{C}$. Anopheles mosquitoes were identified based on their appearance and collected in the evening using a suction tube. They were kept in a nylon net cage and starved for 4 hours before testing. A filter paper in water was placed in the cage overnight. The cage test, a quick and cost-effective method to assess mosquito repellency, utilized a $30 \times 30 \times 30$ cm acrylic cage [13,23&24]. Room temperature was maintained at $25^\circ\text{C} \pm 20^\circ\text{C}$ with 60-70% humidity. For the experiment, one side of the cage had a control sample, and the other two sides had mosquito-repellent printed fabric. The cage bottom was half-filled with repellent fabric and half with control. Thirty mosquitoes were released, and their resting positions were observed at 15, 30, 45, and 60 minutes [21,25&27]. The mosquito-repellency percentage was calculated using the formula:

Mosquito Repellency (%) = Number of specimens on controlled fabric / Total exposed specimen in cage $\times 100$.

Results and Discussion

According to the Debye Scherrer formula, the average grain size obtained from the biogenesis of calcinated nanoparticles was 8.9 nm, as shown by the XRD data. Utilizing a particle size analyzer, the average diameters of the pre-calcinated and post-calcinated (200°C) TiO_2 nanoparticles were found to be 31.8 nm and 17.8 nm, respectively. FTIR spectra of basil (TiO_2) nanoparticles showed both forms of aromatic ring vibrations and O-H stretch. Pre-calcinated nanoparticles revealed the presence of alcohols, phenols, and aliphatic fluoro carbons at various wavelengths, as well as the C-O stretch (1023 cm^{-1}) of terpenoid components. Basil nanoparticles were analyzed morphologically using a scanning electron microscope (ZEISS EVO MA-50), showing particle diameters of 60 nm to 93 nm for calcinated nanoparticles and 113 nm-146 nm for pre-calcinated nanoparticles. Samples containing two types of nanoparticles in varying ratios showed different colours when printed. A modified cage test was used to determine the effectiveness of these samples as mosquito repellents.

Table 1: Effectiveness of fabrics coated with nanoparticles extracted from Ocimum basilicum (leaves) at repelling mosquitoes

Samples	Before post treatment	After post treatment
BC-10	93.00 ^a	90.25 ^a
AC-10	91.50 ^a	85.25 ^b
AC-5	84.50 ^b	82.25 ^{bc}
BC-5	83.75 ^b	79.75 ^c
C	75.25 ^c	69.75 ^d
	F-value=37.80***	F-value=52.42***
	CD=5.32	CD=4.26

Samples with the same superscript letter (a, for example) have statistically equal means, meaning there are no appreciable differences between them. On the other hand, sample means denoted by various superscript letters (a, b, bc, c, d) show a substantial difference between those individual samples.

*** indicate the significance at 0.1% level of significance

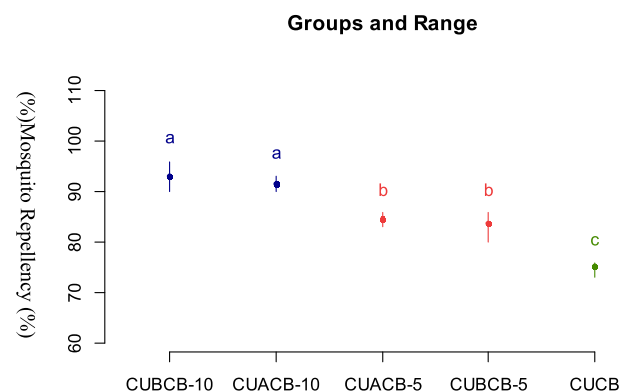


Fig. 1: Mosquito repellent percentages of samples before post treatment with Ocimum basilicum (leaves) nanoparticles [samples that had the same letter are statistically similar, while samples that had different letters had significant differences.]

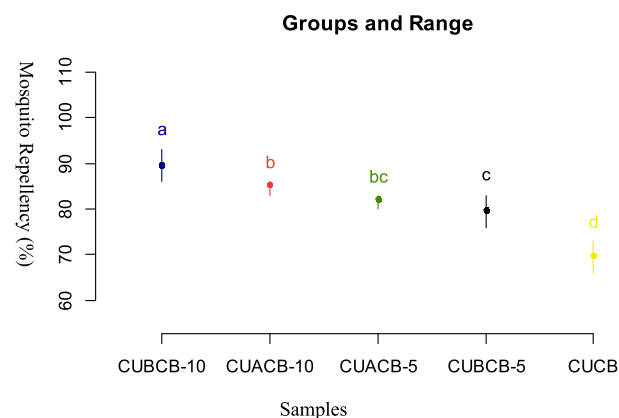


Fig. 2: Mosquito repellent percentages of post treated samples with Ocimum basilicum (leaves) nanoparticles [samples with same letter are statistically equivalent, and sample with distinct letters have significant differences]

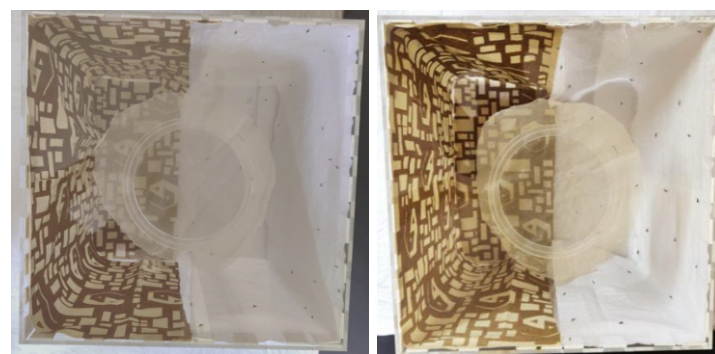
Fig.1 and Table 1 show the findings of a modified cage test used to determine the mosquito repellent efficiency of the samples. The efficacy of fabrics printed with *Ocimum basilicum* (leaves) nanoparticles as a mosquito repellent is significantly higher than that of the control sample, both before and after post-treatments, at less than 1% level of significance. The analysis of variance (ANOVA) table for the Before post-treatment reveals significant differences among the fabric types (F-value = 37.8075, $p < 0.001$). The response variable, BP (presumably mosquito repellent efficacy), shows that the fabric types have a substantial impact.

Furthermore, data in table 1 show that samples recorded 93.10% mosquito-repellent efficacy prior to treatment, with 10% of printed samples containing calcinated nanoparticles coming in second. The calcined and pre-calcined samples printed with 5% nanoparticles had an efficacy of 84.50% and 83.75%, respectively. At the less than 1% threshold of significance, these samples differ significantly from the control sample but not from each other which is also represented in Figure 2. This may be due to the active surface area due to the presence of nanoparticles.

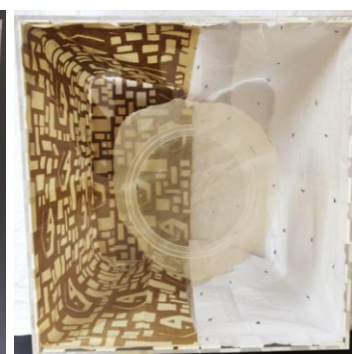
Upon testing and noting the repellence percentage post-treatment, the samples containing 10% pre-calcinated nanoparticles exhibit an efficacy of 90.25%. The results showed that samples printed with 10% pre-calcinated nanoparticles were 85.25% effective, whereas samples printed with 5% calcinated nanoparticles were 82.25 percent effective at repelling mosquitoes and 79.75 percent effective with 5% pre-calcinated nanoparticles. The control sample's effectiveness in keeping mosquitoes away was 69.75 percent. The samples printed with 10% calcinated nanoparticles are significantly different from the 10% pre-calcinated nanoparticle sample, according to the one-way analysis of variance shown in Table 1. All the samples printed with nanoparticles are significantly different from the control at less than 1% level of significance. Similarly, samples printed with 5% nanoparticles differ markedly from samples with 10% pre-calcinated nanoparticles. Post treatment, every sample has shown a decrease in the effectiveness of the insect repellent. Additionally, samples printed with 10% pre-calcinated nanoparticles showed a 2.75 percent decrease in mosquito repellent efficacy, whereas samples printed with 10% calcinated nanoparticles showed a 6.25% efficacy decline. There was a 2.25% and 4.0% decrease in the effectiveness of mosquito repellent in samples printed with calcinated and pre-calcinated nanoparticles 5%, respectively. In contrast, the control samples lost 5.5% of their efficacy after post treatment.



CUCB



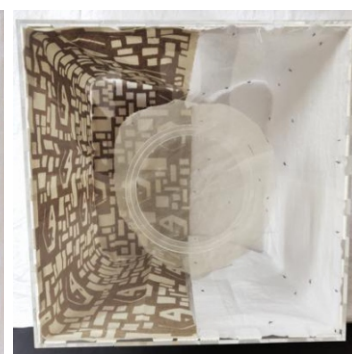
CUBCB-5



CUBCB-10



CUACB-5



CUACB-10

*Figures represented above were observations taken at different intervals

Fig.3 Mosquito repellent efficacy of post treated samples with *Ocimum basilicum* leaf nanoparticles

Table 2: The ability of textiles coated with *Ocimum basilicum* (leaves) nanoparticles to repel mosquitoes following washing cycles

Samples	Means	Wash cycles	Means
BC-10	66.83 ^a	1	81.4 ^a
AC-10	64.83 ^a	3	62.8 ^b
BC-5	55.50 ^b	5	50.6 ^c
AC-5	52.83 ^b	10	37.4 ^d
C	35.33 ^c	15	22.4 ^e
	F-value=44.48***		F-value=121.84***
	CD=5.54		CD=6.07

Samples with the same superscript letter (a, for example) have statistically equal means, meaning there are no appreciable differences between them. On the other hand, sample means denoted by various superscript letters (a, b, c, d, e, etc.) show a large variation between those individual samples.

***indicate the significance at 0.1% level of significance.

The CD value is the same for samples and wash cycles as the MSE (mean square error) was obtained using two-way ANOVA with five samples as treatments and five wash cycles as blocks.

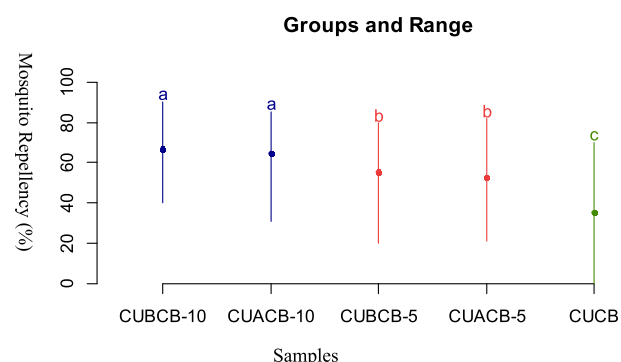


Fig.4: Percentages of mosquito repellency of samples during wash cycles.

[samples with same letter are statistically equivalent, and sample with distinct letters have significant differences]

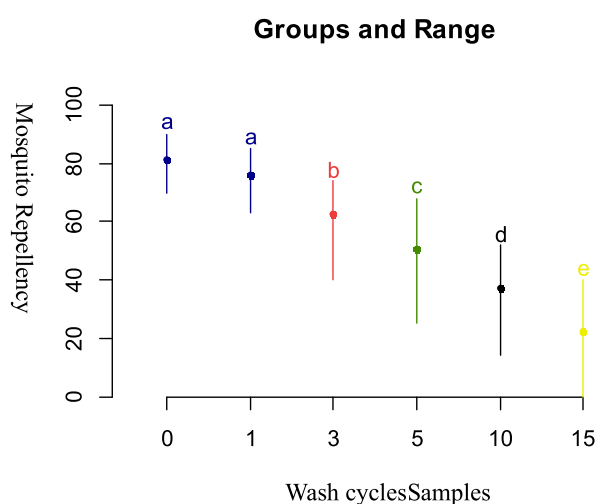


Fig. 5—Percentage of Mosquito repellency among different wash cycles. [samples with the same letter are statistically equivalent, and the sample that has different letters differ significantly]

The efficacy of the mosquito-repelling fabric samples is evaluated by a cage test conducted over a period of fifteen wash cycles, and the wash durability of the prints is evaluated. At less than 1% level of significance, the two-way analysis results show that the samples printed with nanoparticles have significantly higher mosquito repellency than the control sample. During 15 wash cycles, the samples printed with 10% pre-calcinated nanoparticles exhibited the highest repellency, averaging 66.83%. These samples were followed by 10% post-calcinated, 5% pre-calcinated, and 5% post-calcinated samples, and control samples, which had respective repellencies of 64.83%, 55.50%, 52.83%, and 35.33%. Figure 4 further supports this, showing that the control sample continues to have far less repellence than the other samples. Table 2's results also show that there is a highly substantial decline in the percentages of mosquito repellent following each wash cycle, with samples only retaining an average of more than 50% repellency until the fifth cycle. The notable variation in mosquito repellency between the wash cycles is confirmed by Figure 5. The outcomes align with the research conducted by Karthigeyan and Kumar, who observed that samples treated with microencapsulated lemon grass oil had a 10% decline in mosquito repellent efficacy after three hand washes, followed by samples of lavender and thyme oil, respectively.

The study concludes that, over the course of 15 wash cycles, fabrics printed using only dye extract (control) exhibit a significant decrease in repellence after every wash. Samples printed with 10% post-calcinated and 5% pre-calcinated nanoparticles yielded better results, but textiles printed with 10% pre-calcinated nanoparticles recorded the greatest results that lasted after fifteen wash cycles. The findings unequivocally show that the percentage of nanoparticles utilized directly correlates with the wash durability of treated samples. The loss of active chemicals resulting from pressure and detergent application during washing could be the cause of the repellence percentage's progressive decline with an increase in wash cycles.

Conclusion

The study conducted on mosquito repellent textiles demonstrates a promising approach towards sustainable and effective mosquito control.

Utilizing TiO₂ nanoparticles synthesized from *Ocimum basilicum* (sweet basil) plant extracts, the research achieved significant results in developing mosquito-repellent fabrics. These fabrics, particularly those treated with ten percent pre-calcinated nanoparticles, exhibited repellency rates as high as 90-96%. This efficacy, however, diminished after multiple wash cycles, retaining about 50% effectiveness after ten washes, highlighting the durability challenges.

The findings emphasize the potential of integrating green synthesis methods with nanotechnology to produce eco-friendly and effective mosquito-repellent textiles. The use of basil leaf extract not only provides a natural alternative to synthetic chemicals but also aligns with the increasing consumer demand for environmentally safe products. While the study points to the necessity of improving wash durability, the overall results are promising for the future of sustainable textile finishes in mitigating mosquito-borne diseases.

Future Scope of Study

The promising results from the present study open several avenues for future research. Further investigations can focus on optimizing the concentration and binding methods of nanoparticles to enhance durability and functionality under diverse environmental conditions. There is potential for exploring a wider range of bio-based nanoparticles and their synergistic effects in multi-functional finishes, including UV protection, antimicrobial, self-cleaning, and mosquito-repellent properties. Additionally, long-term wearability studies, ecological impact assessments can also be explored.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this research article.

Acknowledgement

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