



ISSN: 2348-5906
CODEN: IJMRK2
IJMR 2020; 7(4): 31-38
www.dipterajournal.com
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Received: 16-05-2020
Accepted: 18-06-2020

Dr. Prathyusha Kantheti
Teaching Associate, Department
of Apparel and Textiles, College
of Community Science, PJTSAU,
Saifabad, Hyderabad,
Telangana, India

Dr. I Rajitha
Associate Professor, Department
of Knitwear Design, National
Institute of Fashion Technology,
Madhapur, Hyderabad,
Telangana, India

Dr. Alapati Padma
Retired Professor & Head,
Department of Apparel and
Textiles, College of Community
Science, PJTSAU, Saifabad,
Hyderabad, Telangana, India

Corresponding Author:
Dr. Prathyusha Kantheti
Teaching Associate, Department
of Apparel and Textiles, College
of Community Science, PJTSAU,
Saifabad, Hyderabad,
Telangana, India

Development of Eco-friendly mosquito repellent printed textiles with synthesized *Ocimum basilicum* leaf dye extract

Dr. Prathyusha Kantheti, Dr. I Rajitha and Dr. Alapati Padma

Abstract

Due to rapid urbanization, climate change and other factors, the ill effects of mosquito bites are growing day by day. The use of repellents such as lotions, coils and liquidators are limited in their efficacy due to various reasons. This has necessitated the development of mosquito repellent fabrics especially in eco-friendly ways as the consumers are moving towards environment friendly products. Eco friendly mosquito repellent fabrics protect the humans from the bite of mosquitoes and thereby promising safety of the environment. On the other hand, today, nanotechnology is advertently used for the development of protective textiles with multifunctional properties. The unique and new properties of nanomaterial have attracted not only scientists and researchers, but also business due to huge economic potential. Hence, the present research was undertaken to develop mosquito repellent printed textiles. The research paper presents the findings of pre-treated cotton fabric screen printed with natural dyes in combination of nanoparticles in two forms (pre calcinated and calcinated) and in two percentages (5% and 10% respectively) using alum as a mordant. The performance of treated fabrics for mosquito repellent efficacy, wash durability along with geometrical properties was assessed. The findings indicated 93 percent mosquito repellent efficacy with ten percent pre calcinated nanoparticles and further the efficacy reduced with increased wash cycles. A very slight change in the geometrical properties like fabric weight, thickness and count was also observed.

Keywords: Mosquito repellent fabrics, *Ocimum basilicum* leaves, synthesized nano particles, cotton fabric, printing, colourfastness

1. Introduction

Mosquitoes are more epidemic in warm and humid places around the world. *Aedes*, *Anopheles* and *Culex* are the most significant species in transmitting mosquito – borne diseases like Zika, dengue, malaria, filariasis, etc to the society. According to the World Health Organization (WHO), more than 1 million people die every year due to mosquito borne diseases and majority of deaths are due to malaria ^[1]. In recent years, the rate of infection through mosquitoes has risen dramatically, and a growing number of scientists are now concerned that global warming will translate into an explosive growth of mosquito-borne diseases world-wide. Methods used in preventing the spread of mosquito borne diseases include vector control, disease prevention using drugs and vaccines and prevention of bites using insecticides, repellents and nets ^[2]. These repellents which were developed and tested will remain effective only for a few months as mosquitoes become increasingly resistant to them ^[3]. Moreover, synthetic repellents were proven to have adverse effect on human skin as well as respiratory systems. Traditionally, many substances have been used in repelling mosquitoes and the most common of them are plant extracts, oils, smoke, etc. Now-a-days, people are looking for safe and eco-friendly repellents, preferably herbal based ^[4]. Mosquito repellent textiles are one of the best means to protect ourselves against mosquitoes without having any adverse effects such as headache, breathing problems and skin irritation, which occur commonly as a result of long-term usage of synthetic repellents. Several plants growing in the vicinity that are often considered as weeds ^[5] may contain essential oils and compounds which were found to be effective insect repellents. Since time immemorial, only a few ethnic groups have been able to tap into the potential of these weeds.

Today, nano systems are designed for the development of protective textiles with multifunctional properties. The unique and new properties of nanomaterial have not only attracted scientists and researchers, but also businesses due to their huge economic potential. On the

other hand, natural dyes are gaining market place amidst growing awareness among the people and an interest towards natural dyed and printed garments and products. For many years, scientists have been investigating natural dyes and their usability in textiles specially in imparting special finishes like deodorizing/aroma [6], insect-repellent [7], flame retardant [8], protection against UV rays [9]. Though there are chemical finished fabrics with built in insect protection (treated with permethrin) specially in the form of nets available in the market, not much work has been done related to organic repellents in the form of prints on textiles. The present research study was an attempt to develop eco-friendly mosquito repellent printed textiles using *Ocimum basilicum* leaves in combination with nanotechnology specially suitable for furnishings and to test the effect of laundering on durability of mosquito repellent finishes.

2. Materials and methods

The main objective of this research was to develop screen printed textiles with mosquito repellent finish by combining nanoparticles with eco-friendly dye sources. The fabric selected for the study was 100 per cent cotton. The fabrics were scoured and printed using natural dye sources in combination with nanoparticles (synthesized from same plant source) for imparting a mosquito repellent finish. The selection of materials and methods or procedures related to development of eco- friendly mosquito repellent printed cotton fabric in combination with natural dyes and to study the wash durability performance of mosquito repellent printed fabrics is discussed below:

Type of research: Experimental research

Dye Source: *Ocimum basilicum* leaves

Nanoparticles: Nanoparticles prepared from *Ocimum basilicum* leaf extract.

Precursor for nanoparticle preparation: Titanium dioxide

Textile substrate: Desized and scoured 100 percent cotton plain weave fabric with 122 GSM and 200 thread count

Pre-treatment: Dried myrobalan fruit powder

Mordants: Alum

Gum used for printing: Cassia tora gum

2.1 Preparation of nanoparticles

Green synthesis or biosynthesis method was employed to synthesize highly stable and well characterized nanoparticles as it was proven to be effective and environmentally friendly substitute to chemical and physical methods [10]. Titanium dioxide was used as a precursor material because of its eco-friendly, hydrophobic, self-cleaning and easily dissolvable nature. The nanoparticles were prepared in two forms - pre-calcinated nanoparticles and calcinated nanoparticles (200°C for 2 hrs). Nanoparticles before calcination have amorphous structures and few impurities on the surface along with plant

compounds. Calcination of nanomaterials is one of the ways to make nanomaterials crystalline, as crystalline materials exhibit better properties in comparison with their amorphous phase in terms of dispersion and removes impurities present on the surface of nanoparticles. On the other hand, calcination step removes the bioactive or phytochemicals present in the nanoparticles when they are exposed to higher temperatures (more than 200°C) [11]. As not enough work has been done on these lines, the experiment was carried out with an aim to investigate the effect of pre and post calcinated nanoparticles on mosquito repellent property. Hence, nanoparticles were used in both forms in two concentrations of 5% and 10% respectively.

2.2 Characterization of nanoparticles

The obtained nanoparticles were analysed for crystalline structure and average crystalline size using Bruker D8 X-ray diffractometer (XRD). The average particle size was measured using HORIBA SZ-100 particle size analyser (PSA). The shape and size of nanoparticles in both the forms was observed using Scanning Electron Microscope (SEM) and Fourier transform infrared spectroscopy (FTIR) analysis was carried out.

2.3 Pre-treatment of fabric with myrobalan

Cotton fabric does not have inherent affinity for most of the natural dyes. Hence desized fabrics were treated using 5 per cent myrobalan solution. Myrobalan contains tannic acid which helps in fixation of dye to the fabric. Solution was prepared by soaking 20g of myrobalan powder for every 100 grams of the fabric for 4 hrs with a material to liquor ratio of 1:30. The fabric was soaked overnight and was dried in direct sunlight. The side of the material exposed to sun was used for printing.

2.4 Extraction of dye from selected plant source

Standardised extraction and printing procedures for cotton were adopted from the research findings on printing with natural dyes by AICRP Scientists of ICAR, Hyderabad. The shade dried basil leaves were weighed, washed, soaked overnight and boiled for 45 minutes in water with M:L:R of 1:10 at 80°C. The extract was then filtered and condensed to 40% (w/w) aqueous extract, which was further used for printing.

2.5 Gum preparation

Dried seeds of cassia were milled into flour and used for preparation of binding agent with M:L:R of 1:20. The prepared solution was boiled until it reached the consistency of a thick paste which was further filtered and used in the preparation of a print paste.

2.6 Preparation of print paste

Print paste was prepared by adding dye to gum in 4:3 ratio to which 10 percent of alum was added according to the weight of the print paste. Nanoparticles were mixed with the prepared print paste in 5 and 10 percent's in two different forms (pre and post calcinated) separately. Five different print pastes were prepared in total as shown in table 1.

Table 1: Coding of print pastes

S. No.	Codes	Print pastes
1.	ACB	Only print paste
2.	ABCB 5%	Print paste + 5% before calcinated nanoparticles
3.	ABCB 10%	Print paste +10% before calcinated nanoparticles
4.	AACB 5%	Print paste +5% calcinated nanoparticles
5.	AACB 10%	Print paste +10% calcinated nanoparticles

2.7 Screen printing and post treatment

Hand screen printing method was employed for impregnating mosquito repellent print paste on to pre-treated cotton fabric. The designs for screens were developed based on the extent of design coverage and suitability furnishing material. Selected cotton fabric was screen printed with the developed mosquito repellent print paste in two strokes with hand squeeze. The printed cotton fabric was then shade dried for 2 days and steamed at 100⁰c using an autoclave. This process helps nanoparticles to adhere well and penetrate the dye into the fibre, thus improving the fastness properties. The fabric was then soaked in 5% NaCl solution with MLR of 1:20 for half an hour followed by washing in luke-warm water with 2 gpl neutral detergent solution to remove the excess dye and nanoparticles on the surface of fabric. The fabric was then rinsed thoroughly under running water and dried in shade.

2.8 Assessment of mosquito repellent property

The mosquito repellent property of the samples was tested using modified WHOPEs excito chamber test method (Fig 1.0). Female Anopheles mosquitoes were identified based on morphological characteristics and were collected during the evening hours with the help of suction tube. They were put in a cage made of nylon net. All mosquitoes were starved of blood and sugar for 4 hours before the tests. A small filter paper wrapped inside the beaker containing water was kept

inside the cage overnight. Laboratory tests were performed during day light hours only. Observations were taken after 10 and 30 minutes of exposure and repeated four times. After each test was completed, the number of escaped specimens to chamber enclosing untreated fabric and those remaining in the chamber holding treated fabric was recorded separately from each exposure chamber.

Escaped specimens and those resting in the chamber with treated fabric, were held separately in small holding containers with food and water in order to note mosquito mortality.

**Fig 1:** Modified WHOPEs excito chamber (Original)

The efficacy of mosquito repellency was calculated using the following formula

$$\text{Efficiency of mosquito repellency (\%)} = \frac{\text{No of specimen escaped} + \text{No of specimen dead}}{\text{No of specimens exposed}} \times 100$$

The mosquito repellent efficacy of the printed fabric samples was tested after 1st, 3rd, 5th, 10th and 15th wash cycles. The samples were even tested for mosquito repellent efficacy before and after post treatment.

2.9. Assessment of geometrical parameters

The prepared and conditioned test specimens were even tested for change in geometrical parameters like fabric weight, thickness and fabric count. Fabric count of mosquito repellent printed fabric was determined using BIS No. 1963-1969 test method. The weight of fabric is described as weight per unit area ^[12]. The weight of a known size of fabric specimen was measured using a sensitive balance as per BIS No. 1964-1970. Heal's thickness gauge was used to measure the thickness of a fabric.

2.10 Durability performance - wash durability testing (AATCC 124-1996)

The samples were washed for 45 min with 5g/l neutral soap solution in distilled water with a MLR of 1:50 at 50±2⁰ C and dried. All samples were tested for the retention of mosquito

repellent activity until 15 washes using standard procedures. The results were compared with the results of unwashed fabric sample.

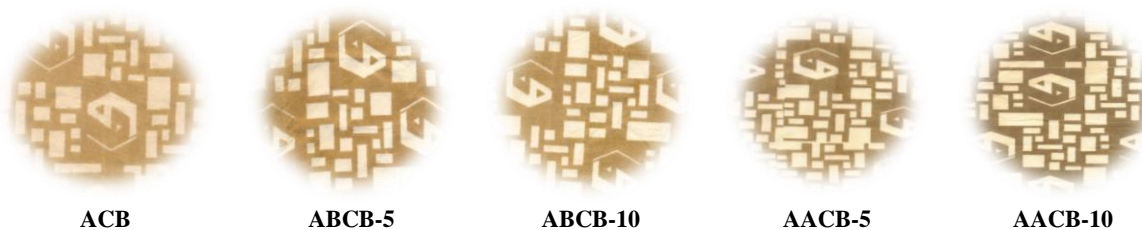
3. Results and discussion

3.1. Characterization of nanoparticles

From XRD analysis, average crystalline size of the nanoparticles sample was calculated as 18nm before calcination and 20nm for after calcination. The results of particle size analysis revealed that the average nanoparticle size of the after calcination was 20nm and before calcination was 32nm. SEM analysis results revealed that, the nanoparticles showed irregular particle structure and the size of the nanoparticles was reported to be 100-120 nm in case of before calcination and size of nanoparticle ranged from 80 nm-90 nm in case of after calcinated nanoparticles respectively. The results of FTIR revealed the formation of TiO₂ nanoparticles. Linalool, Naphthalene, Dodecane, Estragole, Tetradecane, are the biological compounds which were found to act as insect repellent compounds present in basil leaves revealed through GC-MS analysis ^[13].

Table 2: Coding of the Fabric Samples

ACB	Fabric printed with dye from basil leaf extract using alum as mordant (without any kind of nanoparticles)
ABCB 5%	Fabric printed with dye from basil leaf extract using alum as mordant + 5 per cent pre-calcinated basil nanoparticles
ABCB 10%	Fabric printed with dye from basil leaf extract using alum as mordant + 10 per cent pre-calcinated basil nanoparticles
AACB 5%	Fabric printed with dye from basil leaf extract using alum as mordant + 5 per cent post-calcinated basil nanoparticles
AACB 10%	Fabric printed with dye from basil leaf extract using alum as mordant + 10 per cent post-calcinated basil nanoparticles

**Fig 2:** Colours obtained by samples printed using dye in combination with nanoparticles (original)

Colour change in the samples was observed with the addition of nanoparticles in different forms and in different percentages as shown in Fig 2.

3.2 Mosquito repellent efficacy of printed samples with *Ocimum basilicum* (leaves) nanoparticles

The mosquito repellent percentages of fabrics treated with *Ocimum basilicum* leaf dye extract in combination with nanoparticles are presented in the below table 3.

Table 3: Mosquito repellent percentages of fabrics printed with *Ocimum basilicum* (leaves) nanoparticles

Printed samples	Samples before post treatment				Post treated samples			
	No of specimen in the cage	No of specimen escaped	No of specimen dead	Mosquito repellency in %	No of specimen in the cage	No of specimen escaped	No of specimen dead	Mosquito repellency in %
ACB	8	22	-	73	9	21	-	70
ABCB-5	3	27	-	90	4	26	-	86
ABCB-10	2	28	-	93	3	27	-	90
AACB-5	4	26	-	86	5	25	-	83
AACB-10	3	27	-	90	4	26	-	86

*No of specimen exposed = 30 nos.

The mosquito repellent fabrics treated with *Ocimum basilicum* (leaves) dye in combination with nanoparticles, was evaluated for mosquito repellent efficacy using excito chamber test method. The results indicated that 93 per cent repellency was recorded by samples printed with ten per cent pre-calcinated nanoparticles followed by ten per cent calcinated and five per cent pre calcinated nanoparticles which exhibited 90 percent efficacy. The samples printed with five per cent calcinated nanoparticles showed 86 per cent efficacy, whereas, samples printed with only dye extract displayed 73 per cent efficacy as shown in Fig 3.0 before post treatment whereas, the same samples showcased reduction in efficacy after post treatment in which only 90 percent efficacy was observed with ten percent pre-calcinated nanoparticle printed samples. The mosquito repellent efficacy reduced by 4.65 percent after post treatment in both ten percent calcinated nanoparticle printed sample and five percent pre calcinated

nano-particle sample respectively. The reduction in efficacy by 3.4 percent was noticed in samples printed using five percent calcinated nanoparticles. The loss of efficacy was even noticed with sample printed with only dye extract after post treatment. The difference in the efficacy before and after post treatment may be accorded to the loss of nanoparticles and dye extract as a part of post treatment.

The results of the experiment indicated excellent repellency specially when nanoparticles were in pre-calcinated form rather than calcinated form which proved that the efficacy reduces with calcination step in nanoparticle preparation.

These findings corroborated with the results of Vasugi *et al.*, (2010) [14] who observed that samples treated with encapsulated lemon grass leaf extract and lemon grass oil by pad-dry-cure method when tested for mosquito repellent efficacy using cage test, displayed excellent mosquito repellent potency.



ACB

ABCB-5

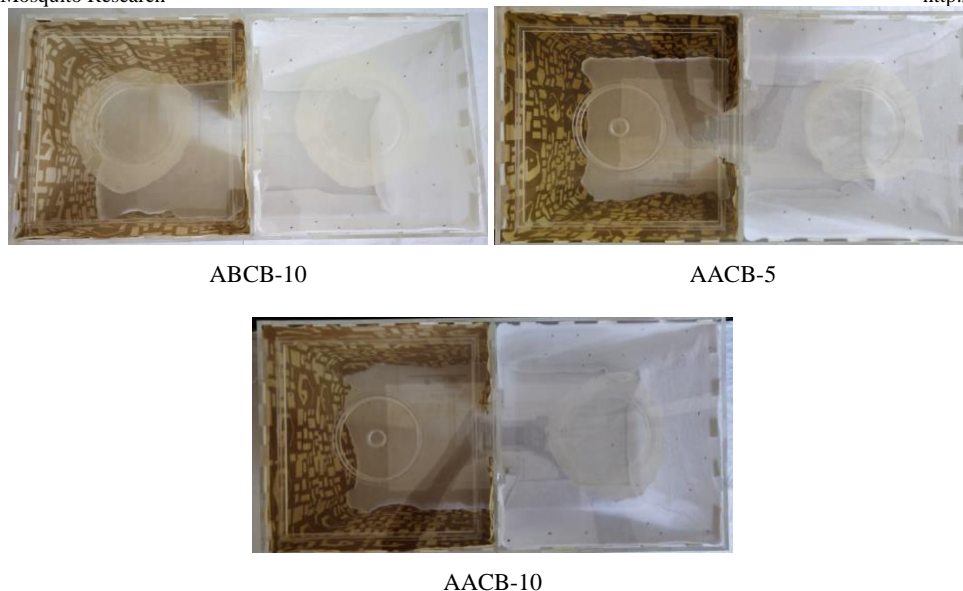


Fig 3: Mosquito repellent efficacy of samples printed with *Ocimum basilicum* leaf nanoparticles.

3.3 Wash analysis of mosquito repellent printed samples with *Ocimum basilicum* (leaves) nanoparticles

The mosquito repellent potency of printed samples with basil

leaf dye extract in combination with nanoparticles was assessed for wash durability (as shown in table 4.).

Table 4: Mosquito repellent efficacy of printed samples after wash cycles – *Ocimum basilicum* (leaves) nanoparticles.

Fabrics	Wash Cycles	Alum (mordant)	
		Repellency (%)	% loss/gain in repellency
ACB	0	70	-
	1	63	-10
	3	46	-34.2
ABCB-5	0	86	-
	1	80	-6.98
	3	70	-18.6
	5	63	-26.75
	10	45	-47.67
ABCB-10	0	90	-
	1	83	-7.77
	3	76	-15.55
	5	66	-26.66
	10	53	-41.11
AACB-5	0	83	-
	1	76	-8.43
	3	66	-20.48
	5	56	-32.53
	10	43	-48.19
AACB-10	0	86	-
	1	83	-3.48
	3	70	-18.60
	5	63	-26.74
	10	50	-41.86
	15	36	-58.13

The wash durability of the printed samples with *Ocimum basilicum* leaf dye in combination of nanoparticles when assessed using excito chamber test method, revealed that fabrics treated with ten per cent pre and post calcinated nanoparticles showcased maximum efficacy of 83 percent followed by 5 percent pre- calcinated nanoparticle printed sample with 80 percent efficacy after first wash. The sample printed with post calcinated nanoparticles in 10 percent displayed 76 percent repellency whereas the control sample displayed only 63 percent efficacy.

The repellency percentage of each sample reduced with

increase in wash cycles. The samples printed with 10 percent pre and post calcinated nano particles retained their efficacy until all 15 wash cycles whereas, 5 percent nano printed samples retained their efficacy till 10 wash cycles respectively.

The repellency of the printed samples documented a gradual decrease with increase in wash cycles, which may be due to loss of print along with nanoparticles bonded with fabric structure due to abrasion from the surface. The results were in harmony with the findings of Yuvasri *et al.*, (2016) ^[15] who noted a 10 per cent decrease in mosquito repellent efficacy

after 3 hand washes of samples treated with microencapsulated lemon grass oil to, followed by samples of lavender oil and thyme oil respectively.

3.4 Geometric parameters

3.4.1 Fabric Count

Fabric count of mosquito repellent printed cotton fabrics in warp and weft directions recorded very slight change or a very minimal change before post treatment using basil leaf extracts with nanoparticles in different concentrations as exhibited in table 5. An increase in fabric count was observed

with almost all the post treated samples in warp direction when compared to samples before post treatment and control. Maximum increase in warp count was noted specially with printed samples using nanoparticles at ten per cent concentration. In contrary, a decrease in fabric count was observed in weft direction specially in post treated samples which were printed using nanoparticles in ten per cent concentration. The marginal variation in the fabric count, might be due to the stretching of yarns in weft and warp directions during printing and post treatment process.

Table 5: Fabric count of the printed samples

Fabric samples	Fabric Count- <i>Ocimum basilicum</i>							
	Before Post Treatment				Post treated			
	Warp count	Per cent change	Weft count	Per cent change	Warp count	Per cent change	Weft count	Per cent change
Control	103		93		103		93	
ACB	103	0	93	0	106	+2.91	92	-1.08
ABCB-5	104	+0.97	92	-1.08	105	+1.94	91	-2.16
ABCB-10	103	0	91	-2.16	106	+2.91	90	-3.23
AACB-5	104	+0.97	94	+1.07	105	+1.94	92	-1.08
AACB-10	102	-0.98	93	0	107	+3.88	91	-2.16

*Control is cotton fabric which is desized and scoured with no further treatments.

3.4.2 Fabric weight

A very slight increase or a minimal increase in weight of the fabrics was observed with printed samples exclusive of nanoparticles when compared to control, especially among samples before post treatment as indicated in table 6. Percentage loss in weight was recorded with samples after post treatment when compared to before post treated samples. The increase in weight of fabrics may due to application of

plant extracts, mordants and gum along with the nanoparticles in the print paste. Whereas, the percentage loss in weight of samples after post treatment, may be due to the loss of excess dye present on the surface of the printed samples. The results were at par with the results of Thite and Gudiyawar., (2015) [16] who also noted an increase in weight of treated samples as compared to untreated, in a study on four diverse herbal and chemical repellents.

Table 6: Fabric weight of samples printed with *Ocimum basilicum* (leaves) nanoparticles

Fabric samples	Fabric Weight (g/m^2) – <i>Ocimum basilicum</i> (leaves)			
	Before Post Treatment	Per cent change	Post treatment	Per cent change
Control	122.2			
ACB	128.5	+5.15	125.0	+2.29
ABCB-5	129.5	+5.97	126.5	+3.52
ABCB-10	131.5	+7.61	128.0	+4.74
AACB-5	129.0	+5.56	126.0	+3.11
AACB-10	131.0	+7.61	127.5	+4.51

*Control is cotton fabric which is desized and scoured with no further treatments.

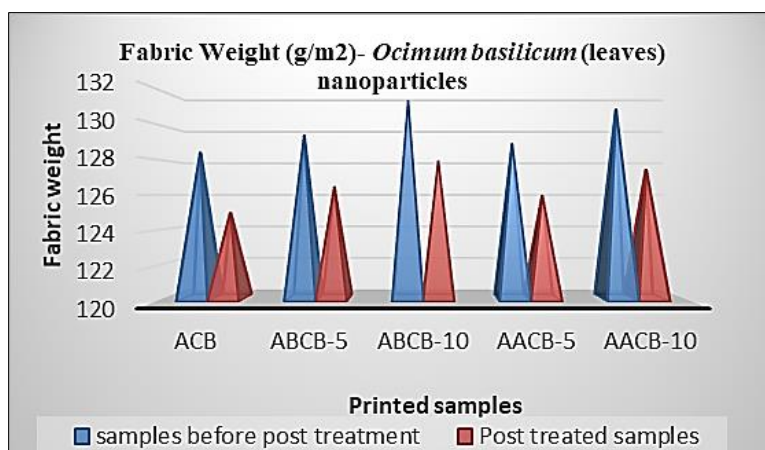


Fig 4: Fabric weight of the samples

3.4.3 Fabric thickness

It is clearly evident from the data in table 7. and figure 5.0 that the thickness slightly increased in all the samples. Among all the samples, those treated with 10 per cent calcinated nanoparticles have displayed higher thickness values followed by pre-calcinated nanoparticle printed samples. The change in thickness observed, can be attributed to the absorption and

penetration of nanoparticles, dye extract along with mordants in to the fiber surface. The results of the study were supported with the results of Sharmila *et al.*, (2015) [2] reported an increase in fabric thickness when experimented on cotton fabric treated with mosquito repellent herbal oil, applied through pad dry cure method.

Table 7: Fabric thickness of samples printed with *Ocimum basilicum* Nanoparticle (Original)

Fabric samples	Thickness in (mm)- <i>Ocimum basilicum</i> (leaves)			
	Before Post Treatment	Per cent change	After Post Treatments	Per cent change
Control	0.27			
ACB	0.31	+14.81	0.30	+11.1
ABCB-5	0.33	+22.22	0.32	+18.51
ABCB-10	0.35	+29.62	0.34	+25.92
AACB-5	0.34	+25.92	0.32	+18.51
AACB-10	0.38	+40.74	0.34	+25.92

*Control is cotton fabric which is desized and scoured with no further treatments.

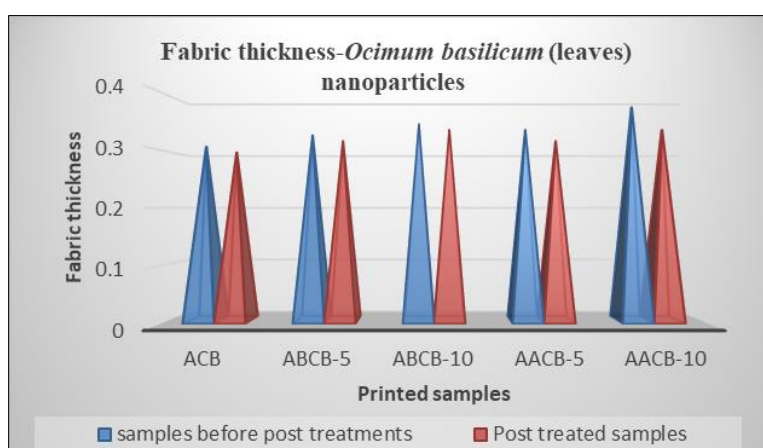


Fig 5: Fabric thickness of printed samples (original)

3.5 Statistical analysis

The statistical analysis of the test results using paired t-test for comparison between before and after post treatments revealed, a significant difference in percentage efficacy with printed samples using alum at 0% level indicating percentage difference in mosquito repellent potency before and after post treatment.

The statistical analysis of the excito chamber test results of samples after wash cycles using F-test indicated that, the difference in mosquito repellent efficacy between the treatments was non-significant, whereas, the difference between wash cycles was found to be significant at 5 per cent level, indicating a definite relapse in efficacy after wash cycles.

4.0 Conclusion

Today in this customer driven market, the products which provide protection by use of natural active compounds derived from the plant extract is a significant revival of interest. The products which are eco-friendly and biodegradable are more acceptable to consumers since they offer the advantage to be non-toxic and eco-friendly. The present study on development of eco-friendly mosquito repellent printed textiles, explored that *Ocimum basilicum* leaf dye extracts can be combined with nanoparticles in order to impart mosquito repellent property in the print form, so as to provide decorative appeal in addition to the functional

performance of fabrics. These fabrics can be used as furnishing materials like curtains in order to avoid the mosquitoes at the door step. The presence of linalool, naphthalene, dodecane, estragole, tetradecane, as biological compounds in the nanoparticles might have helped in the effective mosquito repellency. The study indicated that mosquito repellent property can be imparted by addition of nanoparticles to natural dye extract in print form, so as to reduce man - vector contact.

Limitations of study

- The study was limited to furnishings only.

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