# Development of sustainable aroma and mosquito repellent finish for textiles

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**Abstract**
*Functional finishing of textiles has been in great demand since the last two decades in domestic and industrial applications. Mosquito repellent textiles are one of the most demanded functional finished textiles in the current market. The investigation of this research is to study the combined effect of fragrance and mosquito repellant property on cotton fabric by using Lavender and Citronella essential oil by means of nano encapsulation. Nano encapsulated essential oil prepared via sol gel process and is applied on cotton fabric through nip-pad-dry method. The treated fabric is found to have good mosquito repellant property with reasonable wash durability up to five domestic washes. The aroma release of transformed tissue and washed fabric was estimated by chromatography/mass spectrometry of head space (HS-GC/MS) by measuring the concentration of the main components in tissue treated with essential oils. The results reveal that in each wash cycle, the essential oil is released from the treated fabric as the concentration of chemical compounds decreases gradually. A panel assessment and a field trial of mosquito repellant property have been done to validate the HS –GC/ MS study.*

**Keywords**
*Citronella, HS GC/ MS, Lavender, Mosquito repellant, Nano encapsulation, Sol gel.*

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## 1.0 Introduction:

Aroma-functional textiles such as bed covers, tablecloths, and curtains have witnessed increasing market demand due to their enhanced aesthetic and therapeutic appeal [1]. Among functional textile categories, mosquito-repellent fabrics hold particular significance due to growing public health concerns and the demand for personal protection [2,3]. However, the direct application of essential oils to textiles presents challenges related to their high volatility and limited chemical stability. Aroma delivery in textiles relies on the volatility and sufficient concentration of fragrance compounds, enabling diffusion to the olfactory epithelium and subsequent perception by the olfactory centre in the brain [4–7]. To address the volatility and short-term efficacy of essential oils, nano-encapsulation techniques have emerged as an effective strategy to modulate the release rate of volatile compounds, thereby extending their functional lifespan [8–10]. Nano-encapsulation of aroma compounds using inorganic or hybrid matrices has gained considerable interest for textile finishing. Various fabrication methods for aromatic nano-capsules have been reported in literature [11,12], with the sol-gel technique standing out due to its simplicity, tunability, and suitability for fabric applications.

In this study, a sol-gel encapsulation approach was employed to entrap citronella oil (a known mosquito repellent) and lavender oil (used in aromatherapy for alleviating insomnia), aiming to develop multifunctional cotton textiles with both mosquito-repellent and aromatic properties. The synthesized nano-capsules containing the essential oils were applied onto cotton fabric using the pad–dry method to ensure even distribution and adhesion. To evaluate the wash durability of the treatment, the release behaviour of citronella oil post-laundering was quantified indirectly through analysis of its key volatile constituents using headspace gas chromatography–mass spectrometry (HS-GC-MS). Additionally, the mosquito-repellent efficacy was assessed through a combination of field exposure tests and subjective evaluations obtained from a user panel.

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## 2.0 Material and analytical methods

### *2.1 Materials*

- Lavender oil and Citronella oil purchased from Nishant aromas Pvt Ltd Uttarakhand.
- Fresh lemongrass procured from Mumbai market.
- Tween 80 , Citric acid and Ammonium hydroxide were purchased from Merck India Ltd.
- Ethanol purchased from Gogia and company, Mumbai.
- Tetraethyl orthosilicate (TEOS), Hexadecyl-trimethoxysilane (HDTMS), Methyltriethoxysilane (MTES) were purchased from Sigma Aldrich.
- 100% cotton fabric purchased from Kiran threads, Mumbai.

### *2.2 Equipments*

The following equipments were used in this project work.

Shimadzu QP2020 NX GC MS, Ultrasonic bath, Magnetic stirrer and Oven.

### *2.3 Methods*

*2.3.1 Analysis of essential oil*

Purchased Lavender oil and Citronella oil was analyzed to assess the purity and fragrance components. Chemical components and purity of the essential oil was analyzed by Gas Chromatography Mass Spectrometer. Major Components in Lavender oil were found to be Linalool, Linalyl acetate and lavendulyl acetate. Major components in Citronella oil were found to be Citronellal, Geraniol, Citronellol , Limonene and Citral.

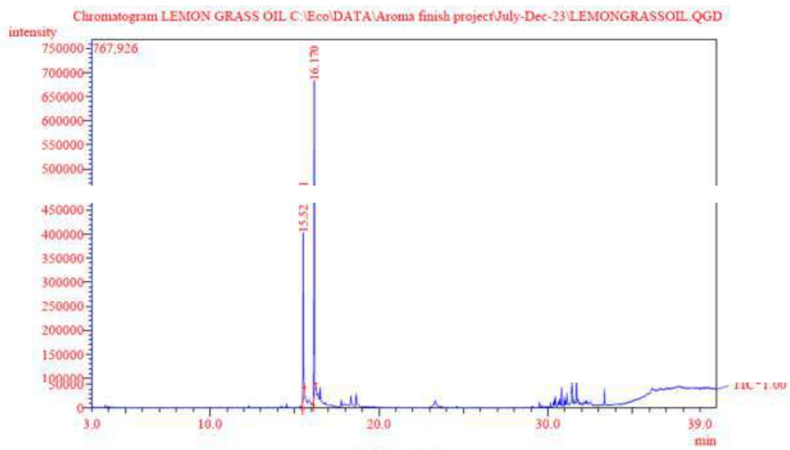
*2.3.2 Distillation of lemongrass*

Fresh lemon grass procured from Mumbai local market (Fig-1) was also steam distilled and the essential oil was recovered by using liquid –liquid extraction with hexane. Steam distillation set up is shown in Fig-2. 2.0 % (yield) pure oil was recovered from fresh lemon grass. GC MS Chromatogram of distilled lemongrass oil and commercial

***Figure 1. Lemon grass collected from local market***

***Figure 2. Photograph of steam distillation set up of lemon grass oil***

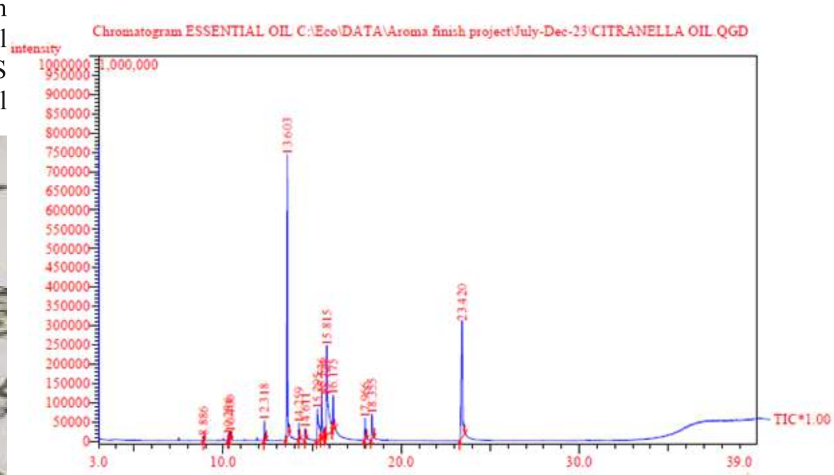
citronella oil is given in Fig 3 & 4 respectively. Purity of the distilled lemongrass oil was found to be better than the commercially available one.




Peak Report TIC

| Peak# | R.Time | Area | Area% | Height | Base m/z | Name |
|---|---|---|---|---|---|---|
| 1 | 15.521 | 934239 | 33.98 | 382920 | 41.05 | Beta Citral |
| 2 | 16.170 | 1815089 | 66.02 | 657228 | 69.10 | Alpha Citral |
| | | 2749328 | 100.00 | 1040148 | | |

***Figure 3. GC MS Chromatogram of lemongrass oil***



Peak Report TIC

| Peak# | R.Time | Area | Area% | Height | Base m/z | Name |
|---|---|---|---|---|---|---|
| 1 | 8.886 | 24855 | 0.36 | 11521 | 93.10 | Beta Pinene |
| 2 | 10.286 | 37823 | 0.54 | 15482 | 119.15 | Para Cymene |
| 3 | 10.400 | 52262 | 0.75 | 21584 | 68.05 | D- Limonene |
| 4 | 12.318 | 134803 | 1.94 | 52658 | 71.05 | Linalool |
| 5 | 13.603 | 2001174 | 28.74 | 734436 | 41.05 | Citronellal |
| 6 | 14.259 | 102904 | 1.48 | 41646 | 71.05 | 4-Terpinol |
| 7 | 14.611 | 77198 | 1.11 | 29010 | 59.05 | Alpha Terpinol |
| 8 | 15.295 | 505279 | 7.26 | 80222 | 41.05 | Citronellal |
| 9 | 15.536 | 394859 | 5.67 | 115152 | 41.05 | Beta Citral |
| 10 | 15.729 | 70695 | 1.02 | 25991 | 93.10 | Linalyl acetate |
| 11 | 15.815 | 1325322 | 19.04 | 233911 | 69.10 | Geraniol |
| 12 | 16.175 | 295048 | 4.24 | 91465 | 41.05 | Alpha Citral |
| 13 | 17.966 | 150903 | 2.17 | 56094 | 69.10 | Neryl acetate |
| 14 | 18.355 | 205452 | 2.95 | 64873 | 69.10 | Geranyl acetate |
| 15 | 23.420 | 1583322 | 22.74 | 303552 | 149.10 | Di ethyl phthalate |
| | | 6961899 | 100.00 | 1877597 | | |

***Figure 4. GC MS chromatogram of citronella oil***

*2.3.3 Application and evaluation of wash durability of Citronella oil & Lavender oil on cotton fabric.*

2.3.3.1 Preparation of sol gel solution

A solution comprising ethanol (200 ml), water (8 ml), and ammonia (10 ml) was added to a 500 ml reactor flask. The experimental setup comprised a reflux condenser, a magnetic stirrer, and a dropping funnel, all assembled on a thermostat-regulated heating mantle. The reaction temperature was consistently maintained at 85 ± 1 °C, while the stirring speed was fixed at 850 rpm to ensure homogeneous mixing. Reagents were introduced dropwise via the dropping funnel to facilitate controlled addition and maintain thermal stability throughout the reaction process. and a dropping funnel. A mixture of 21 ml tetraethylorthosilicate (TEOS) and 13 ml ethanol was added drop wise into the reactor while maintaining the same temperature and stirring speed. The reaction continued at 85 °C for 2 hours. Subsequently, the temperature was increased to 100 °C, and a second mixture comprising 9 ml methyltriethoxysilane (MTES) and 25 ml ethanol was introduced into the flask. The reaction was allowed to continue for 24 hours under the same stirring speed (850 rpm) and temperature (100 °C). At the end of the process, $SiO_2$ nano-particles (nano-$SiO_2$ sol) were obtained. In the sol-gel process, the solution (sol or gel) enables the transition of metal-organic precursors into a solid, often through hydrolysis and condensation reactions, forming an inorganic polymer network. The poly condensation reaction

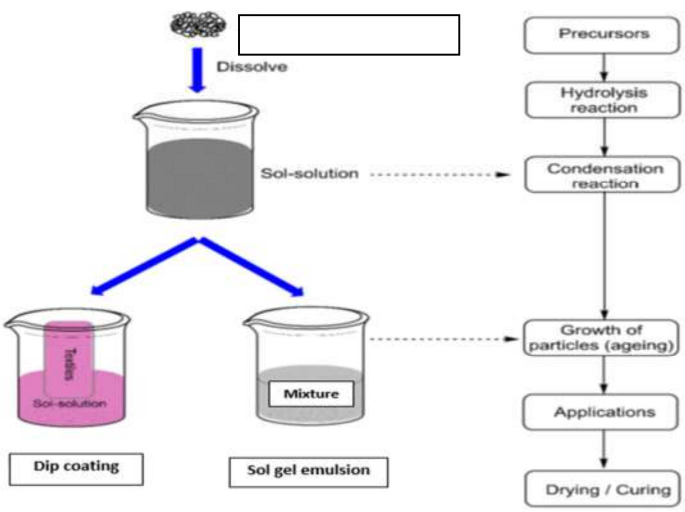



***Figure*** 5. various stages of sol gel synthesis

transforms Si-OR to Si OH – comprising species into siloxane compounds which is the basic chemical principle of sol gel treatment of silica- based material. fig- 5 represents the various steps of sol gel formation.

2.3.3.2 Encapsulation of Essential Oils in Mesoporous Silica

10 % Essential oils (citronella and lavender) were encapsulated into the above prepared mesoporous silica, along with 1 ml surfactant (Tween 80), 20 ml citric acid (1% solution), and 10 ml hexadecyltrimethoxysilane (HDTMS). The mixture was stirred at 850 rpm for 3 hours at room temperature. The encapsulated oil-loaded mesoporous silica was then collected for further characterization and application. Fig. 6 illustrates the physicochemical reaction of nano encapsulated oil in sol gel.

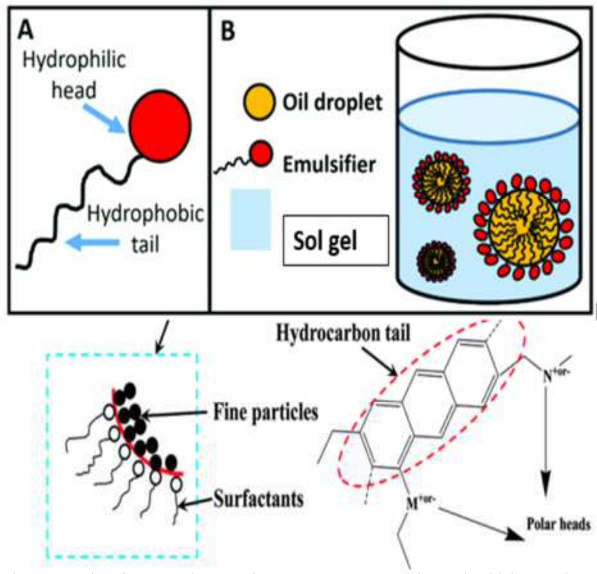



***Figure 6. formation of nano encapsulated oil in sol gel***

2.3.3.3 Aroma Nano-Capsule Wall Material synthesis

The sol-gel process is widely used as a physicochemical method to fabricate nano capsules using metal oxides. It allows precise control over morphology and ensures uniform encapsulation of active ingredients under mild conditions. The general sol-gel encapsulation process consists of four main steps:

1. Formation of an aqueous droplet by mixing a metal oxide precursor, solvent, and surfactants (sol phase),
2. Gelation via precursor polymerization to form an oxide matrix,
3. Addition of the active ingredient (essential oil),
4. Encapsulation of the active agent within the forming matrix.

The chemical reactions of sol-gel and formation of oil nano capsules is given in Fig.7 .

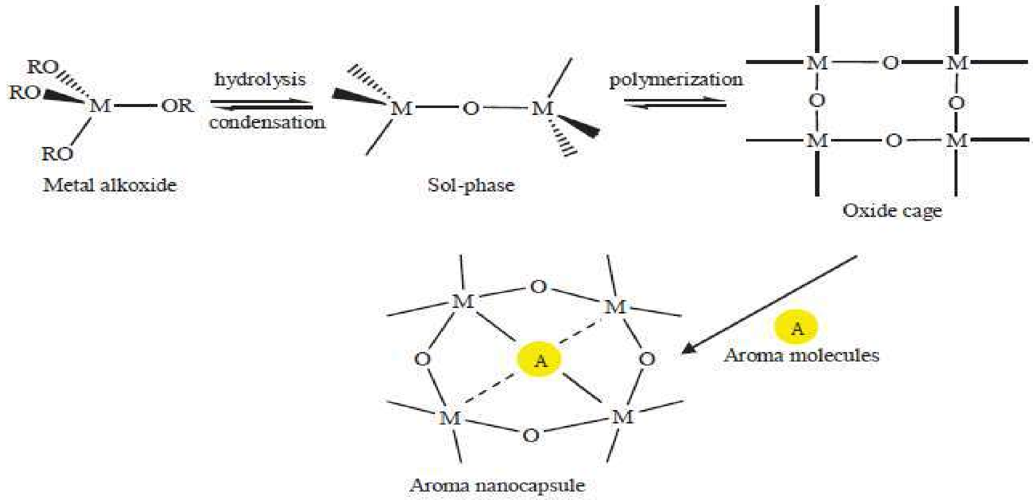



*Figure 7. Chemical reactions of oil sol-gel, and formation of oil nano capsules*

2.3.3.4 Application of nano capsules on to Cotton Fabric

The applications on cotton fabric take place in two stages. In the first stage, synthesized nano-$SiO_2$ sol applied on to cotton fabric by nip–pad–dry-cure method. The fabric was dried at 80oC for 5 min and cured at 130 °C for 5 minutes. In the second stage of application, fabric was treated with encapsulated aroma sol gel by spraying on both sides of fabric by using a spray gun. A final curing step was performed at 110 °C for 2 minutes.

The chemical modification of the fabric occurred in two main stages. In the first stage, when a sol- gel is applied on cotton fabric, the nano – SiO2 particles are deposited on the surface of cotton fabric. A condensation reaction occurs between the hydroxyl (-OH) groups on the cotton fabric surface and the silanol groups of the silica precursor, resulting in covalent bonding of $SiO_2$ to the cellulose matrix. The deposition of silica nanoparticles on the fabric surface effectively reduces surface energy, thereby enhancing the hydrophobicity of the textile material. The presence of methyl (-$CH_3$) functional groups further contributes to the hydrophobic nature of the $SiO_2$ particles. The synergistic effect of the Nano-scale roughness imparted by $SiO_2$ and the intrinsic micro-scale texture of the cotton substrate produces a hierarchical or binary roughness. This dual-scale surface morphology significantly improves the water-repellent properties of the treated fabric by altering its wetting behaviour. In the subsequent step, the sol–gel treated fabric undergoes a surface modification with hexadecyl-trimethoxysilane (HDTMS). In aqueous ethanol, HDTMS hydrolyses to form alkyl silanols, which can further condense with available hydroxyl or silanol groups on the $SiO_2$-modified surface, leading to the formation of a low-energy hydrophobic layer. These react with the surface hydroxyl groups to form a self-assembled monolayer. This treatment locks in the encapsulated aroma (the "active agent") within a core-shell structure, where the oil represents the core or payload, and the $SiO_2$ serves as the protective shell. Upon mechanical triggers like abrasion or friction, the capsule wall ruptures, releasing the aroma into the environment.

**3.0 Results and Discussion**

***3.1 Morphology of the nano capsules***

In order to study the morphology of nano capsules, nano capsules in sol-gel form, treated fabric and washed fabrics were analyzed by Scanning Electron Microscope (SEM). SEM micrograph of nano capsules, treated fabric and five washed fabric is given in Fig. 8, Fig.9 and Fig.10 respectively. From Fig.8 it can be seen that the nanoparticles are varying in size with a size range of 80 nm to 286 nm. Average majority of the particles are spherical in shape with a size of 116 nm. From Fig 9 it can be seen that the oil nano capsules are embedded on the fabric surface. The size of nano capsules are in the range of 104 nm to 612 nm. Higher the size in treated fabric could be due to agglomeration of nanoparticles on the fabric surface. From Fig.10 it can be seen that the washed fabric (after 5 wash) also contains nano capsules on the surface of fabric. This clearly indicates the presence of oil nano capsules on the surface of washed fabric.

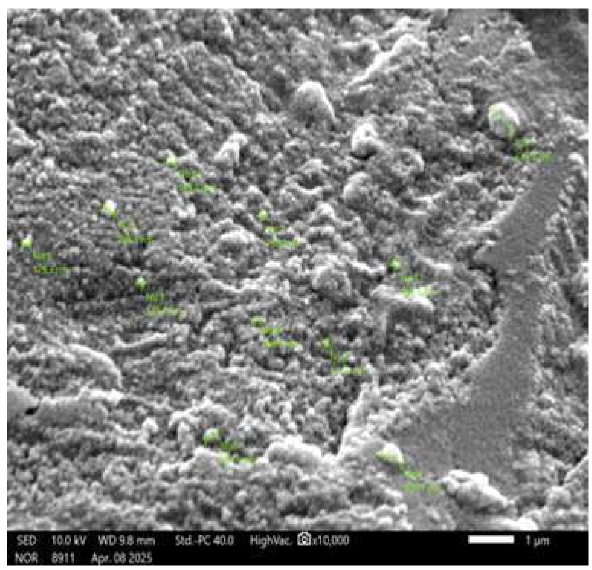



***Fig 8 SEM micrograph of sol gel encapsulated essential oil***

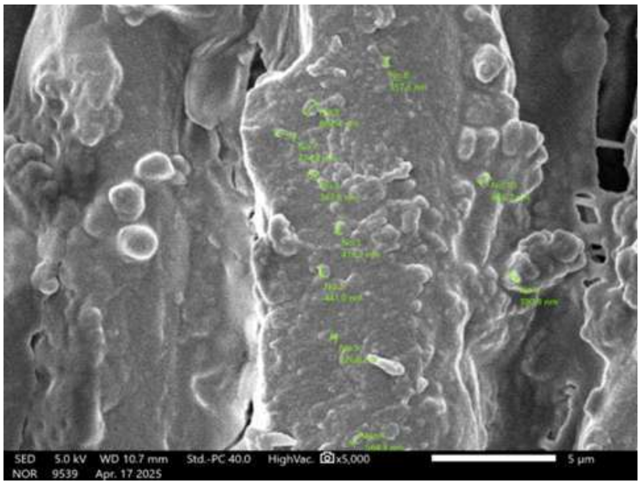


*Figure 8. SEM micrograph of encapsulated oil treated fabric*

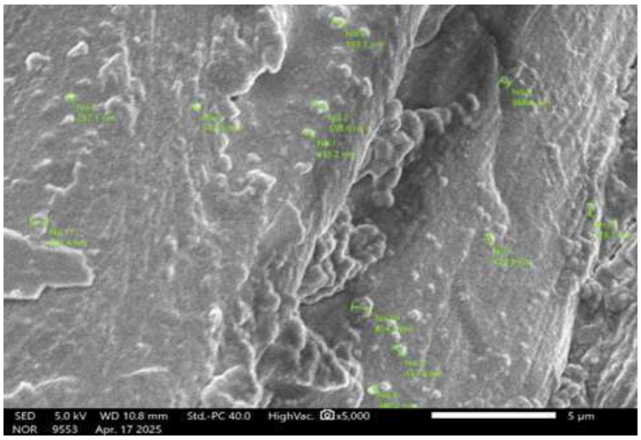


*Figure 9. SEM micrograph of encapsulated oil treated fabric*

### *3.2 Wash durability of citronella oil, lavender oil and mixture of citronella oil and lavender oil treated fabric*

Citronella essential oil, Lavender essential oil and a mixture of citronella oil and lavender oil treated fabric was subjected to machine wash (front loading washing machine) using 1% detergent at 300C for 15 minutes wash-rinse cycle. The qualitative evaluation of fragrance was assessed by a panel of judgment. Treated fabric and washed fabric was given to a 10 member panel for fragrance rating in the order of 0-5 (No fragrance –very strong fragrance). Average rating of citronella treated fabric and washed fabrics is given in Table 1. The average ratings of panel members for treated fabric and one washed fabric is 5 and 4.7 respectively. This clearly shows that treated fabric is durable to one domestic wash. The washing cycle was repeated further four times and asked the same panel members for their rating. Most of the panel members could detect the fragrance in fabric up to 5th wash cycle with an average rating of 1.0. However, after 5th wash cycle most of the panel members could not sense the presence of fragrance in washed fabric. This indicates that citronella oil treated fabric is durable up to 5 washes. Panel assessment rating of lavender treated fabric and a mixture of citronella and lavender treated and washed fabric is given in Table 2 and Table 3 respectively. From Table 2 it can be seen that all the panel members could sense the fragrance up to the 4th washed fabric. Further, in the 5th and 6th washed fabric a mixture of opinions cropped up within panel members. Similarly from table 3 it can be seen that most of the panel members agree with the presence of fragrances in the washed fabrics up to 5th wash. Further, hardly few members could detect the fragrances in the washed fabrics. From the above observations it is confirmed that essential oil treatment is durable up to 5 washes.

*Table 1. Panel assessment for wash durability of citronella oil treated fabric*

| Panel assessor | Treated fabric | After 1 wash | After 2 wash | After 3 wash | After 4 wash | After 5 wash | After 6 wash |
|---|---|---|---|---|---|---|---|
| 1 | 5 | 4 | 5 | 3 | 2 | 1 | 0 |
| 2 | 5 | 5 | 5 | 3 | 2 | 1 | 0 |
| 3 | 5 | 5 | 5 | 4 | 1 | 1 | 1 |
| 4 | 5 | 4 | 4 | 3 | 2 | 0 | 0 |
| 5 | 5 | 4 | 4 | 2 | 1 | 1 | 0 |
| 6 | 5 | 5 | 5 | 3 | 2 | 1 | 0 |
| 7 | 5 | 5 | 4 | 4 | 3 | 2 | 1 |
| 8 | 5 | 5 | 5 | 3 | 1 | 0 | 0 |
| 9 | 5 | 5 | 4 | 3 | 1 | 2 | 0 |
| 10 | 5 | 5 | 5 | 3 | 1 | 1 | 0 |
| **Mean** | 5 | 4.7 | 4.6 | 3.1 | 1.6 | 1.0 | 0.2 |

*Table 2 Panel assessment for wash durability of lavender oil treated fabric*

| Panel assessor | Treated fabric | After 1 wash | After 2 wash | After 3 wash | After 4 wash | After 5 wash | After 6 wash |
|---|---|---|---|---|---|---|---|
| 1 | 5 | 4 | 4 | 5 | 3 | 1 | 0 |
| 2 | 5 | 3 | 4 | 3 | 2 | 1 | 0 |
| 3 | 5 | 3 | 3 | 4 | 2 | 0 | 0 |
| 4 | 5 | 4 | 4 | 4 | 3 | 1 | 0 |
| 5 | 5 | 5 | 2 | 4 | 2 | 0 | 1 |
| 6 | 5 | 4 | 4 | 3 | 2 | 0 | 0 |
| 7 | 5 | 5 | 4 | 3 | 4 | 0 | 0 |
| 8 | 5 | 3 | 3 | 4 | 3 | 1 | 1 |
| 9 | 5 | 5 | 4 | 3 | 2 | 2 | 0 |
| 10 | 5 | 5 | 4 | 3 | 1 | 1 | 0 |
| **Mean** | 5 | 4.1 | 3.6 | 3.6 | 2.4 | 0.7 | 0.2 |

*Table 3 Panel assessment for wash durability of citronella oil and lavender oil treated fabric*

| Panel assessor | Treated fabric | After 1 wash | After 2 wash | After 3 wash | After 4 wash | After 5 wash | After 6 wash |
|---|---|---|---|---|---|---|---|
| 1 | 5 | 5 | 5 | 4 | 2 | 2 | 1 |
| 2 | 5 | 5 | 5 | 5 | 3 | 1 | 1 |
| 3 | 5 | 4 | 3 | 5 | 2 | 2 | 0 |
| 4 | 5 | 5 | 4 | 4 | 3 | 1 | 0 |
| 5 | 5 | 5 | 4 | 3 | 2 | 0 | 0 |
| 6 | 5 | 4 | 4 | 3 | 1 | 3 | 2 |
| 7 | 5 | 5 | 3 | 3 | 1 | 2 | 0 |
| 8 | 5 | 5 | 4 | 2 | 2 | 1 | 0 |
| 9 | 5 | 5 | 4 | 3 | 1 | 1 | 0 |
| 10 | 5 | 5 | 3 | 2 | 1 | 1 | 0 |
| **Mean** | 5 | 4.8 | 3.9 | 3.4 | 1.8 | 1.4 | 0.4 |

### *3.3 HS GC MS analysis of citronella oil, Lavender oil and mixture of citronella oil and lavender oil treated and washed fabrics*

In order to validate the qualitative panel assessment of treated fabrics and washed fabrics, the same have been quantitatively analyzed by HS GCMS. 10 g each of treated fabrics and washed fabrics were heated at 100oC for 30 minutes in a closed headspace vial of 25 ml capacity and 1.0 ml of the headspace vapors were injected into a GC MS as per the following conditions.

GC MS make and model: Shimadzu GC Nexis 2030, MS QP2020 NX

Column : DB5 MS (30mX0.25mmX0.25µm)

Injector temperature: 250$^0$C

Detector temperature: 260$^0$C

Interface temperature: 270$^0$C

Oven temperature: 40$^0$C,(3 min), @ 8$^0$C,200$^0$C.

Column flow: 1.5 ml/min.

Carrier gas: Helium.

Detected components were identified from the built in NIST library. Area percentage of the major components in the treated fabrics and washed fabrics was calculated and is given in the bar chart from Fig.11 to Fig.13. Fig 11 is the bar chart of major chemical components in citronella treated fabrics and washed fabrics. From Fig. 11 it can be seen that the major components in citronella oil treated fabric is limonene (46%) followed by Citronellal (35%), Geraniol (8.0%), Citral (6.0%) and linalool (5.0%). In each wash cycle, the chemical components gradually decrease till 5th wash. Further in the 6th washed fabric only two components were detected which is also below 3.0%. These data are in agreement to the panel assessment of citronella oil treated and washed fabrics.

Fig.12 is the bar chart representing major chemical components in lavender oil treated fabric and washed fabrics. From Fig.9 it can be seen that the major chemical components in lavender oil treated fabric is linalool (44%), followed by linalyl acetate (41%), lavandulyl acetate (2.0%) and limonene (2.0%). At the end of 5th wash, only two components were detected which is linalool and linalyl acetate of 5.0% each. Further in the 6th washed fabric linalool and linalyl acetate detected is below 1.0%. This data is also in agreement with the panel judgments of lavender oil treated and washed fabrics.

Fig.13 is the bar chart representing major chemical components in a mixture of citronella and lavender oil treated and washed fabrics. From Fig.10 it can be seen that major components in the citronella oil and lavender oil treated fabric is linalool (33%), followed by linalyl acetate (32%), Citronellal (16%), Geraniol (3%) and lavandulyl acetate (2%). In each wash cycle the fragrance components diminishes and at the end of 5th wash cycle three major components detected were linalyl acetate (9.5%), linalool (9.0%) and Citronellal (4.0%). However, in the 6th wash cycle, components detected are linalyl acetate (3.0%), Citronellal (1.2%), and linalool (0.3%). This data is also in agreement with the panel judgments.

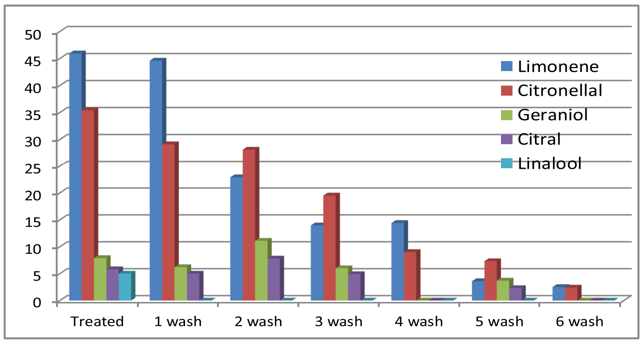



***Figure 11 - Wash durability of citronella oil treated fabric***

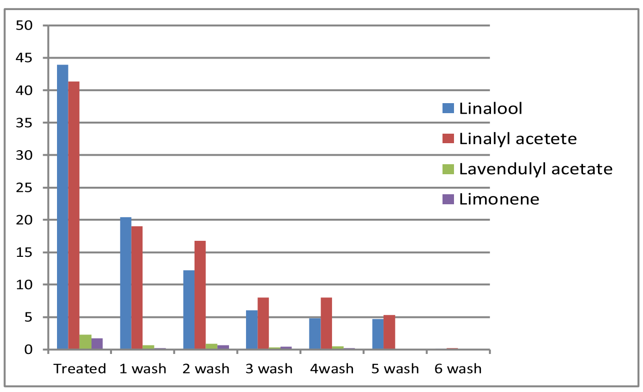



***Figure 12 - Wash durability of Lavender oil treated fabric***

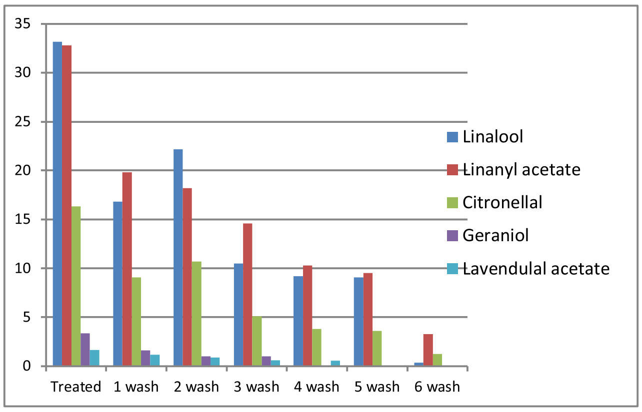



***Figure 13 wash durability of Citronella and lavender oil treated fabric***

### *3.4 Field trials*

User trials are crucial to evaluate overall product performance. 25 treated pillow covers along with pillows were supplied to Janiv Ashram, an old age home situated in Kharad village, Thane district. After using the pillows for one month a feedback was sought from the inmates of the Ashram. Each inmate was asked to fill up the feedback questionnaire about the mosquito repellant property, comfort level, sleep quality, wash durability etc. Majority of the inmates opined that the pillow has a pleasant smell and lasts for a couple of washes. All the inmates agree that while using these pillows no mosquitoes enter the bed room. Few inmates reported that by using these pillows their sleep quality improved. Fig.14A and 14B are the photographs of Janiv Ashram where the field study was conducted.

### 4. Conclusions

Fragrance compounds present in essential oils are inherently volatile, making it challenging to retain their aroma on textile substrates over extended periods. One of the primary difficulties in developing fragrance-emitting fabrics is prolonging the olfactory lifespan of the applied aroma Nano-encapsulation offers a promising solution to this issue by controlling the release of volatile compounds. By incorporating essential oils into nanostructured carriers, the storage stability and wash durability of aroma-functional textiles can be significantly improved. Nano encapsulated citronella oil and lavender oil applied on cotton fabric is found to be durable up to 5 washes. In this study, lavender oil and citronella oil were successfully encapsulated with sol-gel technique. Synthesized Nano capsules were in the size range of 80 nm to 286 nm. These Nano capsules can be applied on to cotton fabric by a simple spray dry process. HS GC /MS analysis of treated and washed fabrics shows treated fabrics are durable up to five laundry washes. The same has been confirmed by panel members by assessing the fragrance. A feedback obtained from the user panel reveals that citronella and lavender oil treated pillow covers have a good mosquito repellent property with a pleasant fragrance. The treated pillow covers are durable up to five washes. The same has been validated by HS GC/MS analysis by measuring the fragrance intensity. Hence, home textiles such as curtains, table cloths, sofa covers, sheets etc. which are not required for frequent laundering may be applied with Nano encapsulated citronella and lavender oil.


***Fig.14 A Janiv Ashram entrance***

***Fig.14 B Residence of inmates***

**Conflict of Interest**

Authors hereby declare that there is no conflict of interest in publication of this paper. All the data generated and presented in this paper is based on this project work and is authentic.

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