Textile Preservation: Intervention of Nanotechnology in Traditional Practices

Executive Summary of the Ph.D thesis

by

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I Introduction

Heritage textiles have played a crucial role in the customs and traditions of people throughout history. These textiles serve as significant archives that represent human history, cultural values, and artistic creations. Traditional textiles are displayed and stored in museums all around the world, including artifacts from archaeological excavations, caskets, carpets, tapestries, and ornamental fabrics, costumes, and robes that are almost a century old. These textiles are usually made of natural fibers that can be either cellulosic or protein in nature, and they undergo deterioration easily. They are unique, fragile, and demand care, preservation, and conservation for them to survive for future generations, especially in tropical countries like India where the temperature and humidity are relatively high. Apart from the physical and chemical degradation, the main causes of biological deterioration are usually excessive moisture, warmth, and food supply, which create the ideal environment for moulds, bacteria, and pests to quickly multiply and cause discoloration, fibre breaking, decline in polymerization, holes, and ultimately complete destruction.

Traditional preservative techniques in India involve using dry herbs and spices such as cloves, cinnamon, camphor, neem leaves, tobacco leaves, tulsi, lavender, and eucalyptus, among others, to repel insects and kill bacteria. The various active ingredients found in herbs and spices such as eugenol in clove, monoterpenes in camphor, nicotine in tobacco leaves, phenolic chemicals in lavender oil, and carvacrol in oregano. These ingredients have biocidal and insect repellant effects, making them effective for preserving textiles against pests. However, the traditional techniques involve layering dried leaves beneath preserved textiles, which requires constant cleaning and inspection, and the active ingredients found in herbs and spices are frequently light-sensitive and cannot be applied directly to a surface or fabric.

To overcome these difficulties, the researcher developed a nanoparticle that were coated on cotton and polyester fabrics using a pad dry cure method. The essential oils such as neem, clove, cinnamon, carom oil, were used to formulate nanoparticles using chitosan as a polymer. This made the oils more stable and provided biocidal and insect repellent properties to the textiles. The essential oils diffuse through the chitosan polymer shell, allowing a regulated release of the active compounds and providing long-lasting protection to the textiles against bacteria, fungi, and insects. The polymer shell also helps to prevent the active compound from oxidation and UV degradation.

The aim was to develop a preservative fabric that will provide a scientific approach to preserving heritage textiles and increasing their lifespan without directly applying any finishes onto them. The developed nanoparticle-coated fabric can be used to cover heritage textiles when in storage, as a lining for flat storage, and as padding on hangers and rollers. It can also be used as a backing or covering material for exhibits, helping to slow down the degradation processes and limit further aging of museum textiles while preserving as much as possible their unique characteristics for now and future generations. This will also help to sensitize current and future generations about traditional indigenous practices of preserving textiles. Textiles have historically played important roles in various cultures, often reserved for ceremonial or specific classes of people. Special textiles, such as quilts and heirloom textiles, hold sentimental value and can be treasured for their memories and representation of past lives. The preservative fabric developed under this study can also be used by individuals at home to preserve their personal collections and heirlooms.

To achieve the intensive exploration and experimental process, the objectives framed for the study are as follows:

1.1 Objectives of the study

1.1.1 To study and understand the preservative practices adopted by textile museums and by individuals at home.

1.1.2 To isolate and identify the microorganisms present on the deteriorated cellulosic and protein fabrics.

1.1.3 To identify and develop nanoparticles using essential oils.

1.1.4 To study and compare the properties of nanoparticles in terms of its particle size, polydispersity index, encapsulated efficiency, loading capacity, retention property, and energy dispersive X-ray.

1.1.5 To determine the minimum inhibitory concentration of the developed nanoparticles and to compare its efficacy using individual and combination of nanoparticles against the selected microbial strains.

1.1.6 Application of the optimized essential nanoparticles on the substrate and to test the microbial and insect repellency.

II. Review of Literature

Literature review is a crucial component of any study based on a research topic. The chapter includes the core information pertinent to the theoretical literature and researches which were studied by the investigator. The data was collected from various secondary sources such as books, thesis, journals, and museums. Primary data was also collected by interacting with museum conservators and curators of Baroda Museum & Picture Gallery Library, Vadodara; National Museum and National Crafts Museum of New Delhi; Calico Museum, Ahmedabad; and Anne Lambert Clothing and Textile Collection of Department of Textiles, University of Alberta. Data was also collected from libraries like Smt. Hansa Mehta Library and the department of Clothing and Textiles library of The Maharaja Sayajirao University of Baroda, Vadodara; Calico Museum Library, Ahmedabad; and Cameron library of University of Alberta. Another significant source of collecting literature was through online books and research papers, and websites.

The following subsections were used to categorize and discuss the review of the literature:

2.1 Theoretical review

2.1.1. Heritage textiles

2.1.2. Deterioration of textiles

   a. Factors assisting the degradation of textiles

   b. Biodegradation of textiles

2.1.3. Preservation and conservation of textiles

2.1.4. Essential oils: Chemical composition, antimicrobial and insect repellent mode of action and its limitations and challenges

2.1.5. Nanotechnology

   a. Application of Nanotechnology for preservation and conservation of textiles

   b. Encapsulation: Methods, their mechanism, and advantages

   c. Application of chitosan as a polymer to develop essential oil nanocarriers

2.2 Research review

2.2.1. Approaches towards textile preservation and conservation

2.2.2. Application of natural agents using encapsulation method for developing antimicrobial and insect repellent properties

2.2.3. Application of chitosan as a polymer to develop essential oil nanoparticles

III Methodology

This chapter deals with the methods, procedure and standards required to develop a finish focused on anti-microbial and insect repellent finish to preserve the textiles. An extensive review and a survey were conducted at several textile museums and with individuals who are textile enthusiastic with their personal textile collection to understand the preservative practices used for the textiles. Later, an experimental approach was taken to understand the microflora environment of a preserved cellulosic and proteinic natured textile, isolation and identification of the microbes on a preserved cotton, wool, and silk fabric was performed. Finally, a finish was developed using two different process containing two-steps, including the microemulsion and ionic gelation method and emulsion and ionic gelation method to develop nano particles which were coated on a fabric with the help of a binder using a pad-dry-cure method . The characterization and surface analysis of the nanoparticles and the coated fabric was conducted to test their structure, properties and its application for preserving textiles against microbes and insects.

Research Design

Phase I

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Studying the preservation practices followed at Museums and by individuals at home

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Content study of herbs and spices employed for textile preservation

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Identifying the microorganisms present on the deteriorated cellulosic and protein textiles

Phase II

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Cotton fabric | Polyester fabric

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Preliminary Data

1. Fabric Count
2. Fabric GSM

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Preparation of essential oil nanoparticles

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Wall material:

Chitosan

Core material:

1. Neem oil
2. Clove oil
3. Camphor oil
4. Carom oil

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Parameters:

- Chitosan %
- Oil %
- Surfactant %
- Crosslinking agent%
- Method of Preparation
- RPM

Analyzing properties:

1. Determining the calibration curve of the essential oil.
2. Assessing the Size of the nanoparticle (Malvern Zetasizer)
3. Determining the Encapsulation efficiency
4. Determining the Loading capacity
5. Determining the Minimum Inhibitory Concentration (MIC) of the nanoparticles against the selected microbial strains

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Application of Nanoparticles on the substrate

- Spraying + Padding Method
- Single and Combination of nanoparticles

Phase III

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Analyzing and comparing of functional compounds for the antimicrobial and insect repellent property and their efficacy during their encapsulation on cotton and polyester fabric

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1. Assessment of Antibacterial activity of the finished cotton and polyester fabric by Parallel Streak Method (AATCC 147)
2. Determination of Antifungal activity: Mildew and Rot resistance (AATCC 30)
3. Insect repellent test (ISO 3998: 1997)
4. EDX analysis of the finished fabrics
5. Determining the physicochemical stability of the nanoparticles over time and storage

Methods and procedure followed in order to elicit the necessary data for objectives are as follows:

3.1 Documenting the preservative practices adopted by textile museums and by individuals at home:

The researcher had visited various museums to collect secondary source data from the Calico Museum in Ahmedabad, Raja Dinkar Kelkar Museum in Pune, National Museum in New Delhi, Crafts Museum in New Delhi, and the Textile Art Museum at the Department of Clothing and Textiles, The Maharaja Sayajirao University of Baroda, Vadodara, and Baroda Museum and Picture Gallery, Vadodara. To conduct the interviews with the textile collectors, conservators, and museum curators, an open-ended questionnaire was created. Additionally, details on the textiles' preservation and conservation methods were acquired from primary data sources.

3.2 Isolating and identifying the microorganisms present on the preserved cellulosic and protein fabrics undergone deterioration:

Selected fabrics included over 60 years old cotton and silk that had been preserved by being wrapped in mulmul in the presence of naphthalene balls. These fabrics were degraded and shredded when pulled apart. A wool fabric that had been stored in a department of clothing and textiles storage area for more than 30 years in the presence of naphthalene balls, had holes in it from moth infestation, was also chosen for the investigation to isolate and identify the bacteria and fungi that had grown on these materials. Three samples of each fabric measuring 10X10 cm that were taken from the three separate locations were used for the isolation of the bacterial and fungal colonies, which was done using a saline solution and suspended on nutrient agar for growth. The developed colonies were subsequently sub-cultured on several agar plates and identified using their morphological characteristics. The entire procedure was carried at the Department of Microbiology, The Maharaja Sayajirao University of Baroda.

3.3 Identifying and developing nanoparticles using essential oils:

The selection of the essential oils was made in accordance with the review and the preliminary information gathered from the interviews conducted with museum curators and private textile collectors. The essential oils used were neem, clove, cinnamon, and carom, were chosen for their antimicrobial and insect repellent properties. Chitosan was used as the wall material/shell due to its great matrix capabilities and antimicrobial and mucoadhesive properties. The nanoparticles were prepared using two methods: emulsification followed by ionic gelation and nano-emulsion followed by ionic gelation. The formulation and process parameters for developing the nanoparticles were optimized.

Different percentages of chitosan solutions (0.5, 1, 1.5, and 2%) were prepared by dissolving chitosan in acetic acid. Different polymer: oil ratios (1:1, 1:2, and 1:3) were prepared separately in 10 ml of solvent suitable for the significant essential oil. Two different types of surfactants, Tween 80 and Poloxamer, were tested in various concentrations (0.5, 0.75, 1, and 2%) for the preparation of nanoparticles using both methods. Different concentrations of the crosslinking agent, Sodium Tripolyphosphate (TPP) (1%, 2%, and 3%) were utilized to fabricate nanoparticles. The stirring rate and stirring time were also optimized.

All the parameters for creating the nanoparticles were developed and optimized at the faculty of Pharmacy, The M S University of Baroda.

3.4 Evaluation of the properties of nanoparticles:

The characterization and optimization of the developed nanoparticles was done on the basis of following methods. First, a calibration curve for the essential oils using Ultraviolet/Visible spectroscopy (UV-Vis spectroscopy) to determine the entrapment efficiency of the oils in the nanoparticles. Then determination of the encapsulation efficiency (EE%) and loading capacity (LC%) of the nanoparticles was conducted by adding a solvent suitable for the essential oil to the nanoparticles, centrifuging the solution, and separating the supernatant to estimate drug loading efficiency using (UV-Vis spectroscopy). The particle size and polydispersity index (PDI) of the nanoparticles were then analyzed using Dynamic light scattering (DLS). The minimum inhibitory concentrations (MIC) of the nanoparticles were also determined using the microdilution method and four bacterial colonies Bacillus cereus, Staphylococcus, Pseudomonas , and Escherichia coli were selected to examine the repelling properties of the developed nanoparticles under the study. To study the surface morphology, the nanoparticles were centrifuged and the pallets were lyophilized to further perform for the scanning electron microscope (SEM) analysis at IIT, Delhi.

3.5 Determining the minimum inhibitory concentration of the developed nanoparticles and comparing its efficacy using individual and combination of nanoparticles against the selected microbial strains:

The two-fold dilution method is considered a gold standard technique of antimicrobial susceptibility testing that is characterized by its accuracy in determination of the minimum inhibitory concentrations (MICs), and the ability to extend the antimicrobial concentration as far as required. The MICs of the developed nanoparticles were performed individually and in combination against the selected gram-positive and gram-negative bacterial strains.

3.6 Application of the optimized essential nanoparticles on the substrate and testing the microbial repellency:

Two substrates were chosen to apply the optimized essential oil nanoparticles on: cotton and polyester fabric. Cotton was selected for its durability and breathability, while polyester was chosen because it is a synthetic material that may hinder the growth of microbes. The best performing dilution nanoparticles were then applied on the selected substrate i.e. cotton and polyester fabric using spraying and pad dry cure method. The nanoparticles made from neem, clove, and cinnamon essential oils at their optimized conditions were used for the application, along with 10% citric acid binder. The fabric samples were dipped in the nanoparticle finish for 15 minutes, passed through a padding mangle, and then cured and air-dried.

To evaluate the antibacterial activity of blank nanoparticle and nanoparticle coated fabrics, the Parallel Streak Method (AATCC 147) was used. The fabrics were placed in contact with an agar surface in separate petri dishes inoculated with *Bacillus cereus, Staphylococcus, Pseudomonas,* and *Escherichia coli.* The effectiveness of the finish was determined by observing the inhibition zone around the samples. The antifungal activity was evaluated using the AATCC 30 method. *Aspergillus fumigatus* was grown on a solid medium, and a spore suspension was used to inoculate an agar medium in a petri dish. The test samples were placed on top of the inoculated agar medium, and then the top of the sample was inoculated with the spore suspension. The inoculated test samples were evaluated and rated based on the presence of macroscopic, microscopic, or no growth.

3.7 Surface analysis of the coated cotton and polyester fabric using Energy dispersive X-ray spectroscopy (EDX)

Triplicate samples of the cotton and polyester fabric samples that had been treated with optimized chitosan nanoparticles containing neem, clove, cinnamon, and a combination of clove and cinnamon were assessed for EDX analysis. This examination was used to establish the presence of the coating on the fabric's surface by recognizing the atoms from the finish. As a result, it demonstrated whether the finish had been absorbed or not by both the cotton and polyester fabric samples. The test was conducted at the NanoFab Lab of University of Alberta.

3.8 Determination of the physicochemical stability of the nanoparticles over time and storage conditions.

This experiment was conducted to assess the changes in the amount of oil trapped in nanoparticles over a period of two months, under different storage conditions. As the fabric will be used for preserving textiles in museums or for textile collectors at home, it is likely that it will be kept at room temperature (25°C ± 2) and 65 ± 2% RH, in an enclosed chamber or drawer, or exposed to an open environment. For this purpose, 2 cm x 2 cm samples of the finished nanoparticle cotton fabrics were evaluated for 1 and 2 months. One set of samples was kept in a closed environment in a large petri dish, while the other set was exposed to an open environment. The amount of oil released from the nanoparticle-coated samples was determined by soaking them in a suitable solvent for 24 hours, and the absorbance was calculated using a UV spectrophotometer and compared to that of the initial sample.

3.9 Determination of resistance to insects using standard ISO 3998:1977

The polyester fabric did not demonstrate any repellency to bacterial or fungal species, indicating that the finish had not been absorbed into the fabric; this is likely due to its hydrophobic nature. EDX analysis further confirmed this, as no presence of nanoparticles was observed on the coated polyester fabric. As a result, the fabric was eliminated from the further test for insect repellency. Therefore, only treated cotton fabric samples that had been optimized with neem, clove, cinnamon, and a combination of clove and cinnamon essential oil chitosan nanoparticles were tested for its repellency against the cigarette beetles.

The test involved placing the treated and untreated fabric specimens of the known mass in contact with started 25 larvae for 14 days in a closed clear container or a jar. The lid or the cover of the container were perforated with tiny holes with the purpose of aeration for the larvae's. The jars were incubating the insects in the box at 25± 2°C, for 14 days and further assessment of the samples for its resistance towards the insects by calculating the weight loss of both the treated and untreated fabric. The fabric was also visually assessed for any damages, holes or death of larvae in them. Periodically inspection of the reaction of the insects to the treated samples was also done, to see if they were repelled by it, comfortable with it, or if the treatment is causing any harm or death.

The test was conducted at the Department of Agricultural and Ecological Entomology, University of Alberta.

IV. Results/ Key Findings

The research was aimed to develop a finish coated fabric focusing on anti-microbial and insect repellent property that can be used to preserve the heritage textiles and thereby slowing down the degradation processes and limiting their further aging.

Phase I of the research covers the following points:

4.1 Documenting the preservative practices adopted by textile museums and by individuals at home:

A common practice for preservation of textiles followed at all the museums and by the textile collector at homes was the use of a small bag made of mulmul fabric stuffed with natural herb and spices like clove, camphor, carom seeds, and cinnamon sticks where the textiles are stored. In addition to this, the textiles were placed on the shelves and drawers, which had a layer of either dried neem leaves or tobacco leaves sandwiched between mulmul cloth. The use of naphthalene balls was also rather typical. Besides, fumigation was yearly done on textiles that were infested by insects at the museums.

4.2 Isolating and identifying the microorganisms from the preserved cellulosic and protein fabrics undergone deterioration:

The bacterial colonies were observed more on cotton fabric as compared to silk and wool fabric. Gram-positive bacteria's like *Bacillus cereus, Staphylococcus aureus* was observed to be high in number, as compared to the gram-negative bacteria like *Pseudomonas.* Fungal colonies observed on all the three fabric were majorly *Aspergillus* species.

Phase II covers the following points:

4.3 Identifying and developing optimized nanoparticles using essential oils:

The selected essential oils under the study were cinnamon oil, clove oil, camphor oil, carom seed oil and neem oil. Both Microemulsion and ionic gelation, and emulsion and ionic gelation method was used to prepare the nanoparticles. The parameters that impacted the formation of the nanoparticles were as follows:

- Effect of chitosan concentration on the amount of entrapped essential oil: Low chitosan concentration was associated with insufficient molecule availability, which reduced entrapment. Less oil entrapment caused phase separation, which resulted in the observation of creaming. Whereas, high chitosan concentration led to increase in viscosity and turbidity of the solution leading to less entrapment due to poor penetration of oil.
- Sodium tripolyphosphate (TPP) concentration on the average size of the nanoparticles: TPP was used as a negatively-charged cross-linker that was added to the nano emulsion to interconnect the positively charged chitosan polymer chains. Different TPP concentration was employed (1, 2, and 3%). Less TPP concentration caused inadequate cross linking, leading to soft wall of the particle (chitosan) whereas high TPP led lysis of the nanoparticles
- Impact of surfactant concentration on the particle size and entrapment of the nanoparticles: For all the essential oils, two different surfactants, Tween 80 and Poloxamer 188, were used at various concentrations (0.5, 0.75, 1, and 2%). Low surfactant concentration resulted in insufficient surface tension reduction, which caused the particles to aggregate, and high surfactant concentration resulted in foaming and increased viscosity, which increased particle size and impairing nanoparticle formation, which had an impact on entrapment.
- Impact of stirring rate (RPM) on the average size and entrapment of the essential oil chitosan nanoparticles: For both procedures, various stirring speeds at various stirring times were employed. Particle size diameter was significantly influenced by homogenization speed. It was discovered that speeding up homogenization greatly reduced the size of the nanoparticles. Low RPM caused the nanoparticles to aggregate and increase in size. Additionally, it was found that rapid increases in homogenization speed were unfavourable because they caused the medium to absorb a lot of energy, which in turn caused the essential oils to evaporate, particle aggregation or particle bursting in some cases, and particle enlargement rather than particle size reduction.

4.4 Characterization of the developed nanoparticle formulation:

- Using the dynamic light scattering (DLS) approach, the hydrodynamic size of the nanoparticles was investigated on Malvern Zetasizer. The average diameter of the nanoparticles comprised of clove essential oil was 294 ± 12 nm, those made up of neem oil were 189 ± 14nm, and those made up of cinnamon essential oil were 226 nm ± 8 nm. While the measurements for camphor oil were 218 ± 16 nm and 349 ± 18 nm, respectively, for carrom oil.
- The highest entrapment efficiency was observed at 1% oil concentration. 78.65 ± 1.28% in neem oil nanoparticles, 73.68 ± 2.35% in cinnamon oil nanoparticles, 51.62 ± 1.56% in clove oil nanoparticles, and 69.27 ± 2.89% in carom oil nanoparticles. The lowest amount of entrapment was 24.31% for camphor oil. This can be due to the fact that camphor oil, which has the lowest density (0.88 g/cm3) compared to other essential oils, can be highly volatile, which may have made it less stable during the preparation process and contributed to the lower percentage of entrapment. Camphor essential oil was therefore excluded at this step.
- The PDI is a measure of the width of the size distribution, where a PDI value close to zero indicates a narrow size distribution, while a value closer to one indicates a broad size distribution. The PDI is affected by various factors, including the preparation method, the properties of the drug and the carrier material, and the processing conditions. Thus, controlling the PDI is crucial to ensure a consistent and uniform particle size distribution, which is important for reproducibility and batch-to-batch consistency. In terms of size distribution, the cinnamon chitosan nanoparticles had the narrowest size distribution, with a PDI of 0.241. The neem chitosan nanoparticles had a higher PDI of 0.364, indicating a broader size distribution. The clove chitosan nanoparticles had the widest size distribution, with a PDI of 0.287. The PDI values indicate the degree of uniformity of the particle size distribution, with a lower PDI value indicating a more uniform particle size distribution. The values obtained for all three types of optimized nanoparticles were relatively low, indicating that the particle size distributions were relatively narrow.
- The surface analysis of the optimized nanoparticles with the higher entrapment efficiency was then lyophilized for performing SEM at IIT, Delhi. The SEM images showed that the nanoparticles formed were spherical in structure with absence of cracks and showed a

formation of continuous wall layer. An example of neem chitosan nanoparticles is shown in the below figure 1.

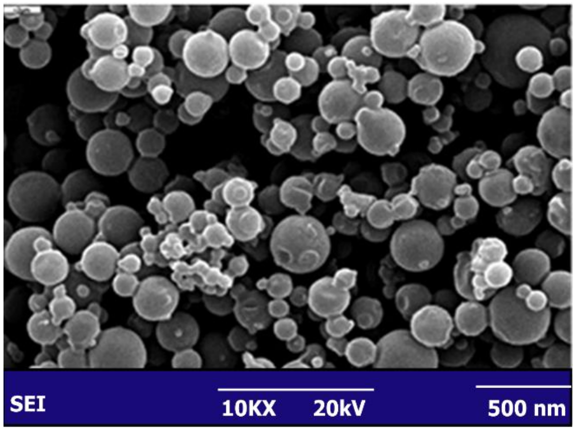


Fig 1: SEM image of Neem essential oil chitosan nanoparticles

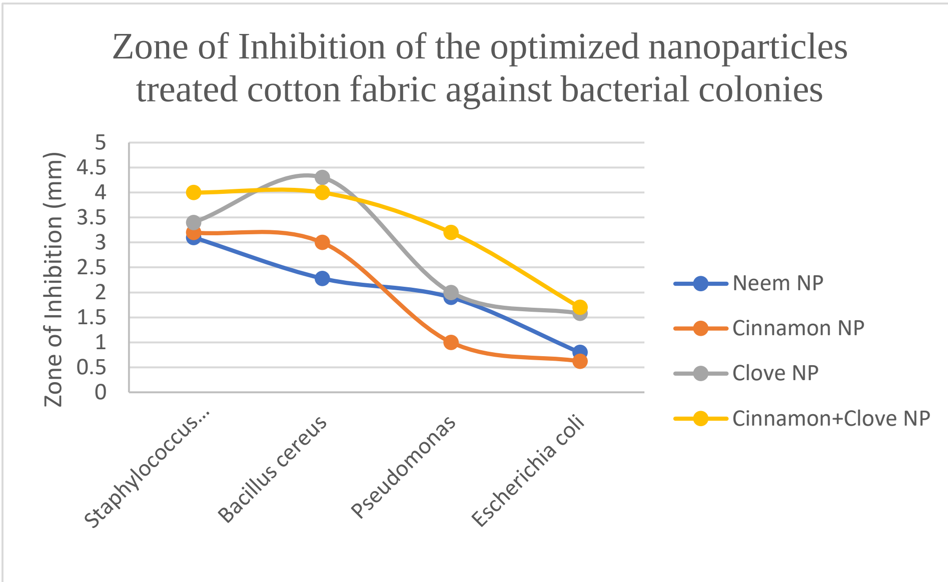
4.5 Determining the minimum inhibitory concentration of the developed nanoparticles and comparing its efficacy using individual and combination of nanoparticles against the selected microbial strains:

MIC of the four developed nanoparticles along with neem +clove and cinnamon+ clove in combination was calculated against two gram-positive bacteria, Bacillus cereus and Staphylococcus aureus, as well as two gram-negative bacteria, Pseudomonas and E. coli, and a fungus, Aspergillus fumigatus. The results showed that all the nanoparticle samples had broad-spectrum antimicrobial activity against both gram-positive and gram-negative bacteria, but there were differences in the MIC values for the different types of nanoparticles. Clove essential oil nanoparticles (C2NPs) were found to be the most effective at inhibiting bacterial growth at a lower concentration compared to other single essential oil nanoparticles, and combining Cinnamon and Clove nanoparticles (C1NPs+C2NPs) enhanced their antimicrobial activity. However, Carom essential oil nanoparticles (C3NPs) did not exhibit any antibacterial or anti-fungal activity, despite having a high entrapment efficiency. Hence, Carom oil was not taken further for the application and eliminated at this step. The study also found that a higher concentration of nanoparticles was needed to inhibit the growth of gram-negative bacteria compared to gram-positive bacteria, which could be attributed to the differences in their cell walls. The differences in MIC values for the different types of nanoparticles were likely due to differences in the types and concentrations of bioactive compounds present in the essential oils used to prepare the nanoparticles. Overall, the study suggests that essential oil chitosan nanoparticles have potential as antimicrobial agents, especially when used in combination.

4.6 Application of the optimized essential nanoparticles on the substrate and testing the microbial repellency:

- The optimized nano particles individual and in combination were diluted in distilled water at the ratios that displayed the best MIC results along with 10% citric acid binder. The application was done by dipping both the selected substrate cotton and polyester fabric in the finish followed by passing it through a padding mangle with 2 dip and 1 nip running at 15m/min, pressure of 15 kgf/cm2, followed by curing at 40C for 5 minutes.
- Fabric samples of 2X2 cm were cut from random places of both the treated fabric to perform antibacterial along with an untreated (controlled) fabric and also with a blank chitosan nanoparticle to see the effect of just chitosan on the antimicrobial behavior and to compare that with the essential oil loaded nanoparticles. The tests were performed in triplicates. Antibacterial test was performed against gram-positive and gram-negative bacterial strains.
- The results indicated that polyester fabric showed no inhibition towards *Bacillus cereus, Staphylococcus aureus, Pseudomonas*, and *E. coli*., regardless of the type of nanoparticle treatment used. This was likely due to the hydrophobic nature of polyester, which hinders the absorption and interaction of the nanoparticles with the bacterial cells.
- On the other hand, cotton fabric showed varying levels of inhibition depending on the type of oil nanoparticle used. As shown in the below graph 1, among the four essential oil nanoparticles tested, a combination of clove and cinnamon oil nanoparticles showed the highest zone of inhibition against *Bacillus cereus, Staphylococcus aureus, Pseudomonas,* and *E. coli.*
- Among the four essential oil nanoparticles tested, combination of clove and cinnamon oil nanoparticles showed the highest zone of inhibition against *Bacillus cereus, Staphylococcus aureus, Pseudomonas,* and *E. coli*. This combination of clove and cinnamon oil nanoparticles showed promising results against these bacterial species, which could be due to their synergistic effect. While in single oil nanoparticles, clove showed the highest zone of

repellency against the four bacterial colonies followed by cinnamon oil chitosan nanoparticles.



Graph 1: Zone of inhibition (mm) of the optimized nanoparticle treated cotton fabric against the selected bacterial colonies

- Clove essential oil nanoparticles which contain high concentration of eugenol exhibited the strongest antibacterial properties, despite having the lowest entrapment efficiencies compared to other essential oil nanoparticles. Despite of the ability of the bacterial repellency, neem oil nanoparticles consistently showed the lowest zone of inhibition against all bacterial species tested which might be the result of its slow-release mechanism explained in the retention study.
- Over all, the study suggested that gram-positive bacteria may be more susceptible to essential oils compared to gram-negative bacteria. In conclusion, the study suggests that clove and cinnamon oil nanoparticles, particularly in combination, showed a synergetic broad-spectrum antimicrobial effect against different bacterial species causing the highest zone of inhibition of 4mm.
- A test for antifungal effectiveness was run on Aspergillus fumigatus. The results for cotton fabric showed no repellency zone, but at the same time, no fungal growth was seen on the

fabric's surface. For antibacterial and antifungal test for the treated polyester fabric showed no resistance. Fungal growth was also observed on the surface of the treated polyester fabric.

4.7 Surface analysis of the coated cotton and polyester fabric using Energy dispersive X-ray spectroscopy (EDX)

- The EDX analysis revealed the presence of Carbon and Oxygen in all the treated samples, along with minor quantities of Nitrogen, Sodium, Phosphorus, and Calcium. One of the examples of clove oil chitosan nanoparticle treated sample is shown in the figure 2.
- The peaks of Nitrogen observed in all samples indicated the presence of chitosan and minerals on the treated fabrics. The trace amounts of Sodium and Phosphorus can be attributed to the use of tripolyphosphate as a crosslinking agent during nanoparticle development. These minerals are likely to be present due to the hydrophilic nature of cotton fabric, which facilitates the absorption and coating of the finish on the fabric.
- The presence of Calcium in small quantities could be indicative of impurities from chitosan, which is a natural polymer used in the nanoparticle synthesis process. However, the amount of Calcium detected is negligible and not significant enough to impact the overall efficacy of the treated fabric. Based on the EDX analysis results, it can be suggested that the essential oil nanoparticles have been successfully applied to the cotton fabric,
- In contrast, the EDX analysis of all essential oil nanoparticles coated on polyester fabric showed no signs of any minerals present on the samples of clove chitosan oil nanoparticles. An example of EDX analysis of clove oil chitosan nanoparticle treated on polyester fabric is shown in figure 3. This lack of mineral presence could be attributed to the hydrophobic nature of polyester fabric, which inhibits the absorption of the finish onto the fabric.
- The lack of impact of the polyester fabric could be attributed to the hydrophobic property of the fabric leading to poor ability to absorb the finish, which was supported by the EDX test. As a result, the polyester fabric was discarded, and the further tests were conducted only on the cotton fabric.

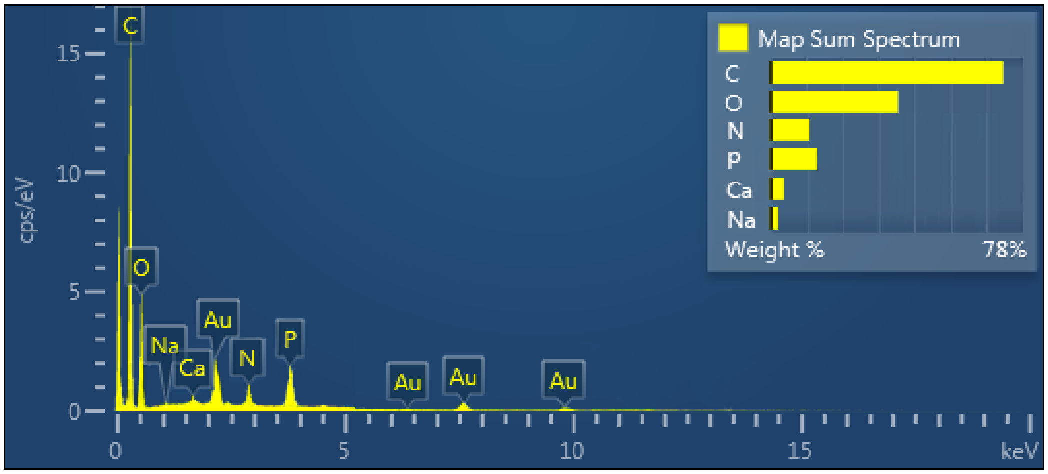


Fig 2: EDX image of clove essential oil chitosan nanoparticles treated cotton fabric conforming the presence of minerals like calcium, sodium, phosphorus and silicon from the chitosan.

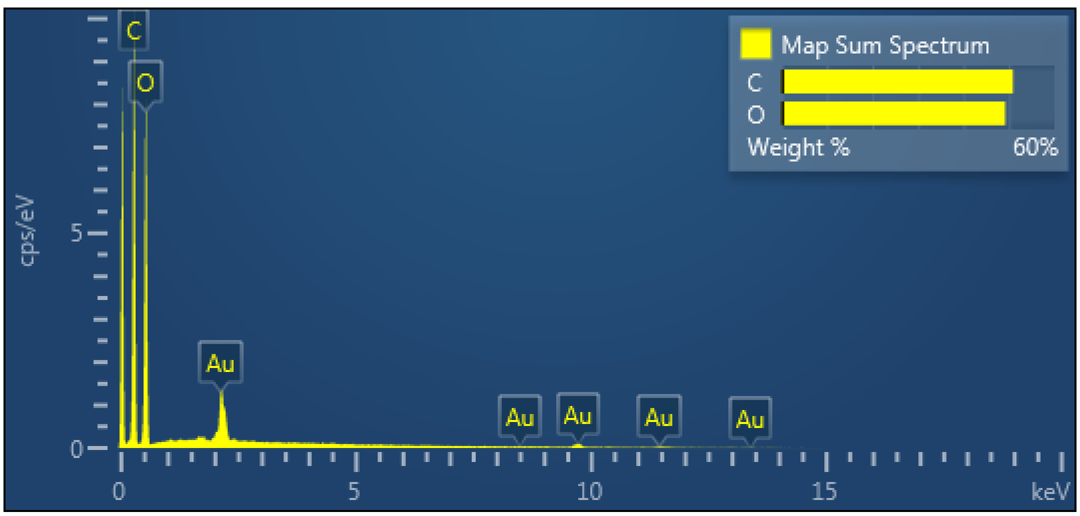


Fig 3: EDX image of clove essential oil chitosan nanoparticles treated polyester fabric showing no traces of minerals

4.8 Determination of the physicochemical stability of the nanoparticles over time and storage conditions.

The *in-vitro* study was done on the nanoparticles that were coated on the cotton fabric substrate at the room temperature in a closed petri dish and in an uncovered petri dish exposed to environment. Keeping the end use of the fabric in mind which will be used in an open environment and in an enclosed environment in a drawer or shelves at a room temperature, the parameters were set accordingly. For analysis, the covered and uncovered samples were soaked in the suitable solvent and the absorbance was calculated using UV spectrophotometer and the percent oil content after one month and two months was compared to zero time (after nanoparticle preparation). The data showed that the fabric treated with neem essential oil loaded nanoparticles exhibited the highest oil retention of 99.10% after one month and 97.32% after two months in a closed environment, followed by clove oil which exhibited the highest oil retention of 98.56% after one month and 97.13% after two months in a closed environment but not in an open environment. Cinnamon oil had the second-highest release rate in an open environment. Neem essential oil exhibited the highest oil retention and this slow-release rate of the oils was found to affect their antibacterial activity, with slower release rates resulting in lower activity. The effectiveness of the treatment decreased over time as the release rate increased, but the percentage of essential oil trapped in the nanoparticle remained above 50% even after exposure to the environment for two months, indicating the durability of the treated fabric.

4.9 Determination of resistance to insects using standard ISO 3998:1977

The treated and untreated cotton fabrics were tested for their ability to repel cigarette beetle larvae, which are frequent insect identified in museums after silverfish and cloth moths. The test was conducted and evaluated in accordance with ISO 3998: 1997. Weight loss or any surface flaws like yarn breaking or holes were not noticed on the treated fabric. However, it was found that larvae exposed to treated fabric perished after four days while those exposed to untreated fabric lived for ten days before dying.

V Conclusion

- It was found that both museums and textile collectors use various methods to preserve textiles, including controlling light, temperature, humidity, and using natural herbs and spices to protect against insects and microbes. However, traditional preservation methods may have limitations and may not be practical for large collections of heritage textiles over an extended period.
- For the proposed study, use of essential oils such as neem, clove, cinnamon, and carom were selected to develop a finish that provides a better preservation environment over a larger surface area for a longer time, resulting in a preservation method that is effective, practical, and has no adverse side effects.
- The dominant gram-positive bacteria isolated from a 60-year-old preserved cotton, silk and wool fabric were Bacillus cereus and Staphylococcus aureus, while Pseudomonas species were the dominant Gram-negative bacteria. Aspergillus was the dominating fungal colony observed on the deteriorated textiles. The study selected these dominant species isolated from the deteriorated fabrics along with *Escherichia coli* to test the effectiveness of the newly developed finish in repelling these colonies.
- Neem, cinnamon, clove and carom essential oil chitosan nanoparticles were prepared using two different methods, the nano-emulsion+ Ionic gelation method and emulsion+ ionic gelation method.
- The size of the nanoparticles developed ranged from 189- 350 nm, and their size distribution was found to be uniform with PDI ranging between 0.241-0.450.
- The highest entrapment efficiency and loading capacity were observed in neem and cinnamon oil nanoparticles with 78.42 ± 1.56% and 74.23 ± 2.11% and 12.28 ± 0.34% and 12.57 ± 0.55%, respectively followed by carom oil with entrapment efficiency of 70.66 ± 2.24% and loading capacity of 11.9 ± 0.64%. On the other hand, clove oil nanoparticles had the lowest entrapment efficiency and loading capacity of 51.62 ± 3.1% and 8.7 ± 0.31%, respectively.
- The MIC results showed that all the nanoparticle samples had broad-spectrum antimicrobial activity against both gram-positive and gram-negative bacteria with Clove essential oil nanoparticles (C2NPs) to be the most effective at inhibiting bacterial growth at a lower

concentration compared to other single essential oil nanoparticles, and combining Cinnamon and Clove nanoparticles (C1NPs+C2NPs) enhanced their antimicrobial activity.

- MIC results of Carom essential oil nanoparticles (C3NPs) did not exhibit any antibacterial or anti-fungal activity, despite having a high entrapment efficiency. Hence, carom essential oil nanoparticles were not carried forward in the study.
- The SEM images of neem, cinnamon and clove essential oil nanoparticles showed that they had a spherical and uniform shape, with absence of cracks, indicating that the wall layer formed continuously during the synthesis process.
- The developed finished was coated on the cotton and polyester fabric selected under the study with the help of a padding mangle using dipping and pad-dry-cure method.
- The antimicrobial studies found that the nanoparticles possess good antibacterial and insect repellent properties and somewhat repellency towards fungi, and can be coated on cotton fabric.
- Clove oil nanoparticle and combination of clove and cinnamon essential oil nanoparticles had the highest antibacterial effect against all the bacterial species, despite clove having the lowest entrapment efficiency.
- The study also found that nanoparticles showed high zone of inhibition towards gram-positive bacteria, when compared to gram-negative bacteria, which could be attributed to the differences in their cell walls.
- The nanoparticles did not show any repellency towards microbes when treated on polyester fabric, possibly due to the hydrophobic nature of polyester which was concluded by performing EDX analysis of the treated fabric as no presence of minerals was observed on polyester unlike cotton fabric which showed presence of sodium, phosphorus, nitrogen and even calcium.
- Neem essential oil loaded chitosan nanoparticles showed the highest oil retention after one and two months in a closed environment, while clove oil exhibited the highest oil retention in a closed environment after one month and in an open environment after two months.
- The slow-release rate of the oils was found to affect their antibacterial activity, with slower release rates resulting in lower activity. However, even after exposure to the environment for two months, the percentage of essential oil trapped in the nanoparticle remained above 50%, indicating the durability of the treated fabric.

- The developed essential oil chitosan nanoparticle treated cotton fabric successfully repelled and caused the demise of Cigarette beetle larvae through prolonged exposure to volatile compounds without causing any damage or loss in weight of the exposed fabric.
- The developed preservative fabric can be used to cover heritage textiles when in storage, as a lining for flat storage, and as padding on hangers and rollers. It can also be used as a backing or covering material for exhibits, helping to slow down the degradation processes and limit further aging of museum textiles while preserving as much as possible their unique characteristics for now and future generations.

Further Recommendations/Suggestions

- The innovative fabric developed through this study is not only suitable for textile museums, but also for heritage museums that contain natural artifacts like paintings that require protection from microbial and insect damage. This widens the scope of the study and offers potential benefits to a wider range of institutions/ private and public sectors involved in heritage conservation.
- Furthermore, the fabric's easy application process makes it accessible to individuals for textile preservation at home. This adds a practical aspect to the study's potential uses, as it offers a simple solution to textile preservation for households.
- In addition to museums and households, the developed fabric's finish can also be utilized in libraries to protect books from insect and microbial damage, ensuring long-term preservation of valuable literature.
- A refill of the developed nanoparticle finish can also be developed for reapplication purposes after the effectiveness of the original coat has decreased. The development of a refill would allow the preservation of textiles to continue after the effectiveness of the initial application has decreased, thereby extending the lifespan of the textiles.
- The nanoparticle finish developed under the study, can be made available in the form of a spray. Further research into the stability and application of the finish may be required to optimize its effectiveness.
- The re-fill solution of this finish can also have various applications in households, such as repelling insects and mosquitoes, can be applied to household bed linens, particularly in hospitals.
- The finish not only has the potential to repel and kill microbes and insects but it also has a natural pleasant odour making acting as an air freshener or an air purifier.
- The potential uses of this natural antimicrobial and insect repellent finish can also extend to car seat covers and public transport like buses, trains, and cabs.
- Overall, the future scope of this study is promising and has the potential to have far-reaching impacts on various industries.

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