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Application of essential oils for finishing of textile substrates

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Abstract

Finishing of various textile fabrics to make it more marketable and its demand is increasing due to global competition, and increasing globalization has created many challenges to the textile researchers and industrialist. The rapid growth in technical textiles and their end uses has generated many opportunities for the application of innovative finishes. The next phase of growth and development of this industry will be focused on three main aspects:

a. Value-added products with enhanced functionality

b. Apparels

c. Sustainable products.

Novel finishing of fabrics has been appreciated by a more discerning and demanding consumer market. With the steady improvement in technology and application standards, a gradual rise has been observed in consumer demands and to reach up to that mark, a manufacturer has to add something to their products to get some added value for their products.

Nano finishing or finishing with macro finishes can provide high efficacy and efficiency for treated fabrics, in comparison to conventional materials, because they possess large surface area and high surface energy that ensures better affinity for fabrics and leads to an increase in durability of the textile functions. Natural oils such as essential oils and other herbal products such as neem oil, castor oil, karanja oil, citronella oil etc. have long been used but today they are also being promoted to be used for finishing application due to their good efficacy without any harmful effects.

The present work deals with the possibilities for application of natural oils on textiles to impart value addition to the substrate. Neem oil obtained commercially was reduced into Nano and micro neem oil emulsion using various types of emulsifiers. The recipe has been attained to get a high add-on on the substrate and also to improve wash fastness properties of the finished samples. The inherent properties of neem oil such as insecticidal, antibacterial, antimicrobial provided the required inspiration to undertake the work and the process used here was ecofriendly as well as neem oil being abundantly available acted as driving force for this work.

The process parameters of preparation and application are relatively easy and feasible and hence can be easily made available to the rural public at much reduced costs and it can create hygiene awareness as well as help in halting the spread of epidemics. These value added products not only produce considerable increase in profit but also build the brand image.

Keywords: value addition, antimicrobial, nano and micro emulsions, technical textiles, combined oil, phase inversion temperature, ultrasonic generators, aliphatic carbon chains, emulsifier, kinetic stability, cotton fabric, polymer surfactant, synergetic surface activity

Abbreviations: PIT, phase inversion temperature; PEG, poly ethylene glycol; CMC, carboxy methyl cellulose; HLB, hydrophile lipophile balance

Introduction

Finishing in the narrow sense is the final step in the fabric manufacturing process, the last chance to provide the properties that customers will value. Finishing completes the fabric's performance and gives it special functional properties including the final touch. In the whole textile manufacturing process from spinning of yarn to finishing of the fabric or garment. Finishing of the textile accomplish 16% of total.

Essential oils are volatile, natural, complex compounds characterized by a strong odor and are formed by aromatic plants as secondary metabolites. An oil is "essential" in the sense that it contains the "essence of" the plant's fragrance- the characteristic fragrance of the plant from which it is derived known for their antiseptic, i.e. bactericidal, virucidal, fungicidal, medicinal and fragrance properties etc. They are used in embalmment, preservation of foods and as antibacterial, analgesic, sedative, anti-inflammatory, spasmolytic and locally anesthetic remedies. Typically, essential oils are highly complex mixture of often hundreds of individual aroma compounds. In nature, essential oils play an important role in the protection of the plants as antibacterial, antiviral, antifungal, insecticides and also against herbivores by reducing their appetite for such plants.[1–3]

The term "vegetable oil" can be narrowly defined as referring only to substances that are liquid at room temperature, or broadly defined without regard to a substance's state of matter at a given temperature. For this reason, vegetable oils that are solid at room temperature are sometimes called vegetable fats. Under the chemical terms they are a combination of triglycerides of higher saturated and unsaturated fatty acids.[4] In other words, these compounds are esters of glycerol and higher fatty acids, containing in their structure long aliphatic carbon chains. These lipids are most commonly extracted from various parts of plants such as seeds, fruits or plant seedlings. Vegetable oils, depending upon the individual percentages of fat acids in the molecule, exhibit a variety of properties.

Figure 1 Neem oil is made of many components. Azadirachtin is the most active. It reduces insect feeding and acts as a repellent. It also interferes with insect hormone systems, making it harder for insects to grow and lay eggs. Azadirachtin can also repel and reduce the feeding of nematodes. Other components of neem oil kill insects by hindering their ability to feed.

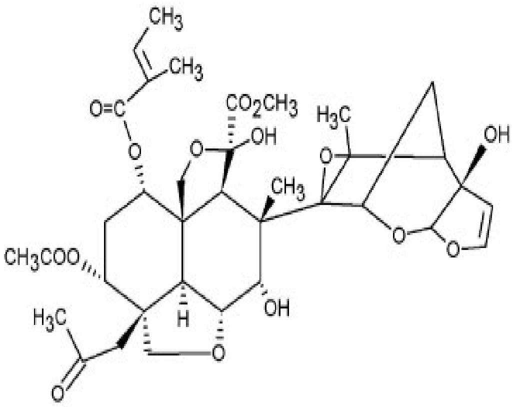

Figure 1 Neem oil and its chemical structure.

Emulsions are a class of disperse systems consisting of two immiscible liquids. The liquid droplets (the disperse phase) are dispersed in a liquid medium (the continuous phase). Several classes may be distinguished: oil-in-water (O/W), water-in-oil (W/O) and oil-in-oil (O/O). To disperse two immiscible liquids, one needs a third component, namely, the emulsifier. The choice of the emulsifier is crucial in the formation of the emulsion and its long-term stability.

Nanoemulsions are submicron oil-in-water emulsions with a Nano scale droplet diameter. They are thermodynamically stable and translucent dispersions of oil and water with a droplet size in the range 100-600 nm. Nanoemulsions are prepared by low-energy methods such as phase inversion temperature (PIT) emulsification, by phase inversion composition or by high shear forces using high-pressure homogenizers or ultrasonic generators.[5,6]

Structure of the emulsion system

A. **O/W and W/O macro emulsions:** These usually have a size range of 0.1-5µm with an average of 1-2µm.

B. **Nano emulsions:** these usually have a size range of 20-100 nm. Similar to macro emulsions, they are only kinetically stable.

C. **Micellar emulsions or micro emulsions:** these usually have the size range of 5-50nm. They are thermodynamically stable.

D. **Double and multiple emulsions:** these are emulsions-of-emulsions, W/O/W, and O/W/O systems.

E. **Mixed emulsions:** these are systems consisting of two different disperse droplets that do not mix in a continuous medium. Several breakdown processes may occur on storage depending on particle size distribution and density difference between the droplets and the medium.[7,8] Nano treated textile applications as shown in Figure 2.

Figure 2 Textile applications of Nano technology.

Emulsifiers

An emulsifier is a substance that stabilizes an emulsion by increasing its kinetic stability. One class of emulsifiers is known as "surface active agents", or surfactants. Detergents are another class of surfactants, and will physically interact with both oil and water, thus stabilizing the interface between the oil and water droplets in suspension. Sometimes the inner phase itself can act as an emulsifier and the result is a nano emulsion, where the inner state disperses into "nano-size" droplets within the outer phase.[8–10]

Role of surfactant in making of emulsion

Surfactants lower the interfacial tension (γ) and this causes a reduction in droplet size. In practice, surfactant mixtures are used and these have pronounced effects on γ and ε. Some specific surfactant mixtures give lower γ values than either of the two individual components. The presence of more than one surfactant molecule at the interface tends to increase ε at high surfactant concentrations. The various components vary in surface activity. Those with the lowest γ tend to predominate at the interface, but if present at low concentrations, it may take long time before reaching the lowest value. Polymer surfactant mixtures may show some synergetic surface activity.[10–12]

Methods of emulsification

Several procedures may be adopted for emulsion preparation and these range from simple pipe flow (low agitation energy), static mixers and general stirrers (low to medium energy), high-speed mixers such as the Ultraturrex, colloid mills and high-pressure homogenizers (high energy) and ultrasound generators (medium to high energy). The method of preparation can be continuous or batch-wise.[11,12]

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Mechanisms of emulsification

A number of different chemical and physical processes and mechanisms can be involved in the process of emulsification

a. **Surface tension theory:** according to this theory, emulsification takes place by reduction of interfacial tension between two phase

b. **Repulsion theory:** the emulsifying agent creates a film over one phase that forms globules, which repel each other. This repulsive force causes them to remain suspended in the dispersion medium

c. **Viscosity modification:** emulsifiers like acacia and tragacanth, which are hydrocolloids, as well as PEG (or polyethylene glycol), glycerin, and other polymers like CMC (carboxy methyl cellulose), all increase the viscosity of the medium, which helps create and maintain the suspension of globules of dispersed phase.[13,14]

Materials and procedures

a. Neem Oil

b. Tween 20 (procured from Mohini organics pvt ltd, India),

c. Single distilled water, Reactive dye, cotton fabric, glauber salt, soda ash, 100% cotton, 100% wool, 100% silk, Polyester/cotton (67/33).

Optimization of neem oil nano emulsion

Initially for optimization purpose 2gpl neem oil was taken with a calculated amount of Tween 20 to formulate a stable nano emulsion. For preparation of emulsion calculated amount of Tween 20 was added to 100ml of distilled water in a 250ml glass beaker with continuous stirring while being kept on magnetic stirrer at room temperature for 15minutes at 100rpm to mix Tween 20 with water without forming. After mixing 0.2g of neem oil was weighed in a glass beaker and added drop wise into this prepared Tween 20 solution with continuous stirring and kept on magnetic stirrer at room temperature for about 4hour at 1250rpm, then this prepared mixture was kept under probe sonication as shown in Figure 3 at 10,000 frequency for different time interval (30, 45, 60min) is indicated in Table 1 and eighteen such formulations were prepared in this way and after completion of visual observation for one week, the best formulation was selected for the further analysis.

Finishing of combine oil nano emulsion by padding technique

2:1:1 ratio of combine oil was chosen for application on fabric. Application of combine oil emulsion (2,4and 6%) was done on cotton fabric. For application 1:1 ratio of combine oil emulsion was taken based on results obtained from the study of combine oil emulsion. Prior to padding, fabric was conditioned at temperature 21°C±2°C and relative humidity 65%±5% for 30min. Finishing solution was prepared according to Table 2. Pre weight of the fabric was taken and dip into prepared solution for 5min before padding, then padding was carried out at 2kg/mt^2 nip pressure and 15mt/min roller speed, two dip two nip with 95% expression, then the sample was air dried and cured at different temperature (60, 70, 80, 90°C) for 120, 90, 60, 30sec respectively for optimizing curing time and temperature. Weight of the fabric after padding was taken to calculate % add-on on the fabric. Eighteen different formulations were prepared for finishing solutions and effect on properties of fabric has been assessed. Based on desired results on cotton fabric best chosen recipe was followed for rest of the substrate as shown in Tables 3-7.

Table 1 Combination of Neem oil with Tween20 in 100ml Distilled water

S. No.	Oil: tween 20	Time of sonication (min)	Average droplet size(nm)
1	1:00:05	45	148
2	1:00:05	60	66
3	1:01	45	114
4	1:01	60	54
5	1:02	45	87
6	1:02	60	46
7	1:03	45	71
8	1:03	60	34

Table 2 Formulation of finishing solution for padding with combine oil emulsion

S.no.	Binder (%)	Resin (%)	Combine oil (%)
1	1	-	2
2	2	-	2
3	-	15	2
4	-	20	2
5	1	-	4
6	2	-	4
7	-	15	4
8	-	20	4
9	1	-	6
10	2	-	6
11	-	15	6
12	-	20	6
13	1	15	2
14	1	20	2
15	1	15	4
16	2	20	4
17	1	15	6
18	2	20	6

Table 3 Quantitative antibacterial analysis test results of DYED cotton fabric on finishing with the nano emulsion

S.no.	No. of washes	% of bacteria reduction	
		With S. aureous	With E. coli
1	0	99.16	97.59
2	5	92.68	90.98
3	10	82.56	76.16
4	15	71.73	65.69

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Table 4 Quantitative antibacterial analysis test results of UNDYED cotton fabric on finishing with the Nano emulsion

S.No.	No. of washes	%of bacteria reduction	
		With S. aureous	With E. coli
1	0	99.91	98.92
2	5	93.69	91.1
3	10	84.7	78.66
4	15	72.87	67.08

Table 5 Effect on tensile strength (kgF) of dyed fabric on finishing with combine oil nano emulsion

S.No.	Fabric	Untreated		Treated	
		Warp	Weft	Warp	Weft
1	Cotton	23.2	21.49	20.26	15.45

Table 6 Effect on K/S of dyed fabric on finishing with the combined oil nano emulsion

S.No.	Fabric	K/S		% Change in K/S
		Untreated fabric	Treated fabric	
1	Cotton	0.7555	0.6787	10

Table 7 Effect on washing fastness of dyed fabric on finishing with the combined oil nano emulsion

S.No.	Fabric	Fastness rating	
		Unfinished fabric	Finished fabric
1	Cotton	4	4-Mar

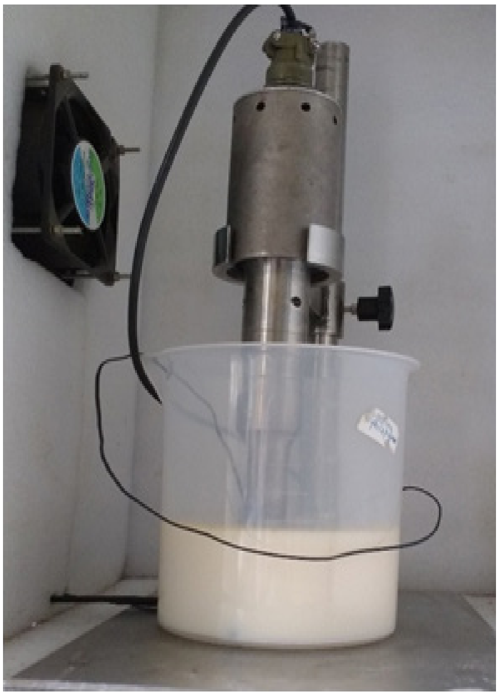

Figure 3 Assembly of ultra sonicator.

Results and discussion

Emulsifiers are an important auxiliary to form emulsions be it water in oil or oil in water. Since oils only stain fabrics, and hence to finish them with efficiently and economically we require emulsions and that too oil in water emulsions, for uniform application and stability. These emulsion finishes have to impart value addition to the substrate after finishing. Hence oils which are basically hydrophobic in nature have to be impregnated on the substrate such that they have good performance properties namely wash and light fastness. Thus emulsion preparation is very necessary to impart good finishing on the substrate.

Preparation of nano neem oil emulsion

At the outset neem oil emulsion was prepared (nano) using tween 20 as an emulsifier and sonicator to form nano emulsion after emulsion preparation its analysis was carried out. Emulsifiers are an important auxiliary to form emulsion i.e. Water in oil or oil in water. Since oils only stain fabrics, and hence to finish them with efficiently and economically we require emulsions and that too oil in water emulsions for uniform application and stability. This emulsion finishes added value to the substrate after finishing. Hence oils which are basically hydrophobic in nature have to be impregnated on the substrate such that they have good performance properties namely wash and light fastness. Thus emulsion preparation is very necessary to impart good finishing on the substrate.

Effect of sonication time and tween 20 concentration on stability of neem oil nano emulsion

Droplet size is an important factor for stability of oil emulsion, droplet size in nano range results in good stability of an emulsion, normally surfactants stabilize oil-in-water emulsion reported that surfactants with lower Hydrophile-Lipophile balance HLB value (3-6) favors the formation of water-in-oil emulsion and surfactants with higher HLB value (8-18) favor the formation of oil-in-water emulsion. Tween20 was opted for making oil-in-water emulsion because of its high HLB value of 16.7, which favors formation of oil-in-water type of emulsion. Due to its high (HLB) value Tween20 molecules diffuse from the organic phase (oil and surfactant) to the aqueous phase and forms low droplet size emulsions. Also being a small molecule surfactant, Tween20 gets adsorbed onto the surface of emulsion droplet more quickly when compared to high molecular weight surfactants (e.g. polymers). Hence, it is more effective in minimizing droplet size than polymeric surfactants.

It was found that on increasing tween 20 (emulsifier) concentrations as well as sonication time reduces the droplet size. As it is indicated in Table 1 the smallest droplet size of the nano neem oil emulsion consisting 1 part of oil and 3 parts of surfactant with 60 min sonication was 34nm.

For reducing droplet size of neem oil, sonication time was found more helpful than surfactant concentration as it can be seen from Table 1. On increasing sonication time from 45min to 60min for the similar neem oil, surfactant ratio gives approximately two times neem oil droplet size reduction, on the other hand on increasing surfactant concentration from 1:0.5 to 1:3 for 45min sonication time resulting in droplet size reduction with the ratio 2, while for 60min sonication time decrease in droplet size was in the ratio 1.9.

Droplet size of nano neem oil emulsion was measured after five

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months storage at room temperature to assess its stability in terms of droplet size, as it can be seen from the (Figure 4a) (Figure 4b). There was no significant effect on droplet size even after five-month storage at room temperature and it was found in nano range only, which shows good emulsion stability with respect to droplet size. 60min sonicated nano neem oil emulsion was stored for this purpose because of its expected longer time stability as it was having low droplet size (in nano range)

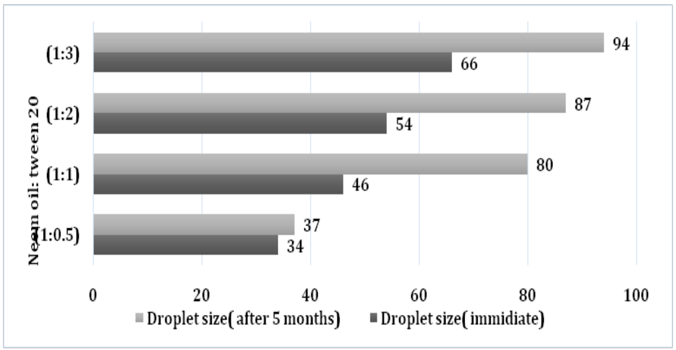

Figure 4(a) Droplet size of neem oil nano emulsions in different time span.

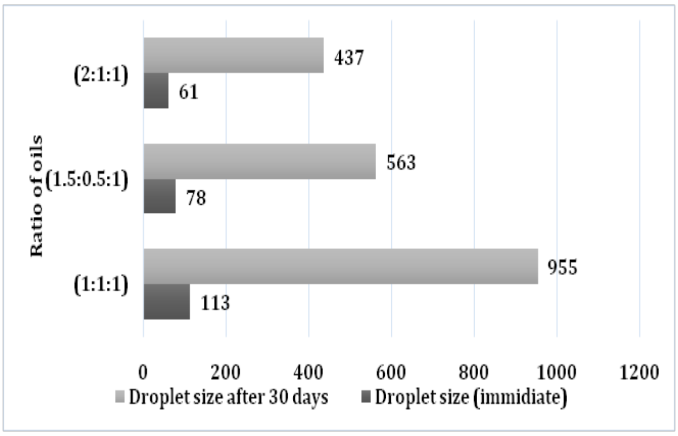

Figure 4(b) Droplet size of combine oil nano emulsion with 1:1 tween 20 ratio.

After 5month storage at room temperature increase in droplet size for 1:0.5 ratio of neem oil and surfactant was approximately 1.4times while in the case of 1:3 ratio increase in droplet size was only 1.08times, which was satisfactory for stability point of view, as immediate droplet size for 1:3 ratio of neem oil and surfactant was 34nm, so agglomeration of droplets were lesser as compared 1:0.5 ratio because immediate droplet size for that was 66nm which was also stable, But immediate and 5month later droplet size of 1:3 nano neem oil emulsion indicates good stability of emulsion. Zeta potential of 1:3 neem oil Nanoemulsions was -43.1mV which indicates good quality of emulsion which means it can be stable for longer time as shown in Figure 5.

The protective aspects of textile have provided the most textile ground for innovative developments. Hygiene has acquired importance in recent years. Odour has become an important factor. Microorganism growth is another factor that has resulted in development of antimicrobial finish. In this project study of essential oil emulsion was carried out to find a utilization of natural sources in place of artificial agents to impart similar effect on the fabric.

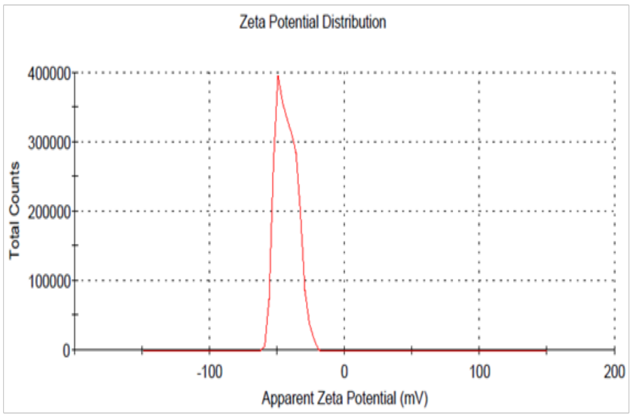

Figure 5 Zeta potential of neem oil nano emulsion (1:3).

Conclusion

Thus the above studies can be summarized as follows

a. Dyed as well as undyed fabric shows up to 99.99% antibacterial property.

b. There were no significance changes seen on colour fastness of the dyed fabric after application of finish. Only 10-15% loss in tensile strength was observed on dyed as well as undyed fabric even after finish application. This is negligible.

c. With the smallest droplet size neem oil nano emulsion along with castor oil was found to be very effective to impart antibacterial finish on the fabric with satisfactory wash fastness.

d. The treated fabric showed good antibacterial efficacy against both *S.aureusand E.coli*. The durability of the antibacterial finish was increased from 5 washes to 15 washes from layer by layer as shown in (Table 3) (Figure 4) application technique with 20 cycles.

Acknowledgments

None.

Conflict of interest

Author declares there is no conflict of interest in publishing the article.

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