A solvent induced crystallisation method to imbue bioactive ingredients of neem oil into the compact structure of poly (ethylene terephthalate) polyester

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Abstract

Neem oil, a natural antibacterial agent from neem tree (\textit{Azadarichthaindica}) has been used to impart antibacterial activity to polyester fabrics. Solvent induced polymer modification method was used and that facilitated the easy entry of neem molecules into the compact structure of polyethylene terephthalate (PET) polyester. The polyester fabric was treated with trichloroacetic acid-methylene chloride (TCAMC) solvent system at room temperature prior to treatment with neem oil. The concentration of TCAMC and the treatment time were optimised. XRD and SEM
results showed that the TCAMC treatment causes polymer modification and morphological changes in the PET polyester. Antibacterial activity of TCAMC pre-treated and neem-oil-treated polyester fabric was tested using AATCC qualitative and quantitative methods. Both Gram-positive and Gram-negative organisms were used to determine the antimicrobial activity. It was observed that the treated fabric registers substantial antimicrobial activity against both the *Staphylococcus aureus* (Gram-positive) and the *Escherichia coli* (Gram-negative) and the effect increases with the increase in concentration of TCAMC treatment. The antibacterial effect remains substantial even after 25 launderings. A kinetic growth study involving the effect of antibacterial activity at various incubation times was carried out.

*Keywords:* Solvent induced polymer modification; Antibacterial; Neem oil; Polyester; Pre-treatment; Trichloroacetic acid.

1. Introduction

Textiles used in hospitals are highly susceptible to pathogenic organisms and they are used as a vehicle to transmit infectious diseases among patients and between patients and medical personnel. Therefore, it is vital to prevent/minimise the propagation of infections and diseases through textile materials used in hospitals and associated environments where hygiene is paramount. For many applications poly (ethylene terephthalate) (PET) is often used as an essential polymer. The improved
functional properties, such as antibacterial property, of PET are vital for a wide range of industrial applications. There is a growing volume of literature demonstrating the survival and growth of microorganisms in textiles and their propagation posed a health risk [1, 2]. In addition, the microbial infestation cannot be eradicated from the fabric even after frequent laundering. In order to combat the above adversities, specific antimicrobial finishes have been developed for application to textiles that contain different fibre types. The major types of synthetic antimicrobial agents that are increasingly used in textiles include triclosan, silver and copper metals and their salts, organometallics, phenols, quaternary ammonium compounds and organosilicons [3]. But these agents would often produce highly toxic or undesirable by-products and their disposability creates environmental concerns. The antimicrobial activity of certain natural dyes has been widely discussed in the literature [4-6]. Effectiveness of various plant extracts against microbes has been studied by a number of researchers in the past [7-9]. Antimicrobial textiles treated with chitosan (deacetylated derivatives of chitin) are also extensively reported in the literature [10-12]. The enhanced antibacterial efficacy of silver nanoparticles [13], chitosan nanoparticle [14, 15] treated textile substrates has recently been published elsewhere. It is established that neem (Azadirachtaindica) possesses active antimicrobial compounds such as azadirachtin, salannin and meliantriol. These compounds are also effective in controlling the insect growth and used as an antifeedent [16]. Studies have reaffirmed the antibacterial activity of neem
constituents such as oil, bark extract and seed extract [17-19]. Recently Prabhakar M. et. al. studied the potential application of neem oil nano-emulsion in aquaculture industry and found that it is an effective antibacterial agent to control the disease against *Pseudomonas aeruginosa* infection in freshwater fish, Labeorohita [20]. The antimicrobial property of neem seed extract on 100% cotton fabric and polyester/cotton blend fabric has been published elsewhere [21, 22]. However, it is difficult to achieve the enhanced and durable antimicrobial property of PET polyester. This is mainly because: semi-crystalline compact structure of PET polymer; and absence of active polar groups within the polymer structure which facilitate cross-linking of antimicrobial agents. Besides, the PET exhibits low surface energy. Several attempts for creating suitable functional groups in PET substrates were made in the past [23, 24]. Most recently, novel pyrazolotriazine and pyrazolylypyrazoloneazo dyes have been synthesized and were applied to polyester by using a disperse dyeing technique, which is commonly used for dyeing polyester, to study the antibacterial activity of the above azo dyes [25]. Subsequently, different approaches have been used by researchers to impart antimicrobial activity to the polyester based textile materials [26-30].

An alternative approach to enhance the antimicrobial effect of polyester is to open up the compact structure of PET polymer by making use of suitable interacting solvents, and this facilitates the easy entry of antimicrobial agents into the compact PET structure. It should be mentioned that the interacting solvents, under suitable
conditions, influence the crystalline and amorphous regions of PET and during this process more voids and cracks are generated within the structure which facilitate the easy entry of antimicrobial molecules into the PET structure and entrap the molecules inside the structure. The interaction of solvents with PET polyester has been the subject of interest for many years because the interacting solvents increase the segmental mobility of the polymer and as a result structural rearrangement takes place (Solvent Induced Crystallisation). When PET polymer is pre-treated with highly interacting solvents, under suitable conditions, internal structure is affected and thereby more voids, cracks are produced which facilitate the entry of bigger size molecules, for instance antimicrobial agents, into the polymer matrix. The extent of PET structural change mainly depends on the concentration of the TCAMC and treatment time. Earlier studies [31, 32] have shown that the interacting power of trichloroacetic acid-methylene chloride (TCAMC) solvent system with the PET is very high and the reagent attacks the polymer matrix, and finally dissolves out the PET at about 25% (w/v) concentration in 5 min duration at room temperature (about 30 °C). This indicates that the solubility parameter of TCAMC reagent is very close to the solubility parameter of PET. It should be mentioned that according to solubility parameter theory the complete dissolution takes place when the solubility parameter value of the solvent and the polymer are close to each other. It is expected that at certain lower concentration of TCAMC treatment the compact structure of PET opens up which facilitates the easy entry of antimicrobial neem compounds.
With the above background in mind, a study was undertaken to produce antibacterial fabric by imbuing the active antibacterial elements of neem into the compact structure of PET. This paper mainly discusses the effect of TCAMC reagent on PET polymer and the incorporation of neem molecules into the PET matrix to achieve the enhanced antibacterial activity for longer duration against both the Gram-positive and the Gram-negative bacteria.

2. Experimental procedures

2.1. Materials used

A plain weave polyester fabric (70 ends × 58 picks) weighing 80 gm\(^{-2}\) was used throughout the study. Laboratory grade trichloroacetic acid (CCl\(_3\).COOH), methylene chloride (CH\(_2\).Cl\(_2\)) and acetone (CH\(_3\).CO.CH\(_3\)) were used. Commercial grade neem oil was used as an antibacterial agent.

2.2 Pre-treatment process

Prior to pre-treatment, FTIR analysis was carried out on 100% TCA, 100% MC and 5% TCAMC to identify the reaction between the two. The fingerprints (Figs 1a-1c) showed that no reaction took place between TCA and MC.

Pre-treatment of the PET fabric samples with TCAMC reagent at various concentrations was carried out in a closed trough at room temperature (~ 30 °C) for 5 min duration. Three different concentrations (1, 3 and 5% w/v) of TCAMC were
used to modify the PET structure. The material-to-liquor ratio was maintained at 1:100 and the contents were shaken manually to ensure uniform treatment. After the treatment the fabrics were rinsed with methylene chloride followed by acetone to remove any adhering reagent on the fabric. The treated samples were squeezed and air dried at atmospheric condition.

2.2.1 Application of neem oil on pre-treated PET

The pre-treated PET fabrics were immersed in 10% (v/v) solution of neem oil, which was previously prepared by using propanol, with material-to-liquor ratio of 1:20 at 80°C for 20 min. After that the fabrics were hot and cold washed thoroughly before subjected them to testing and characterisation.

2.2.2 Determination of dry add-on% of neem-oil-treated PET

The additional weight gain in percent (add-on %) of the 10% (v/v) neem-oil-treated PET was calculated as below:

\[
\text{Dry add-on\%} = \frac{(A - B) \times 100}{B}
\]

Where, \(A\) = dry wt. of neem-oil-treated PET and \(B\) = dry wt. of untreated PET

2.3. Characterization

Instron 4202 (based on CRE principle) (ASTM-D5035-90) was used for tensile testing. The samples (20 cm × 5 cm) were conditioned in the standard
atmosphere of 65% RH, 20 °C, for 24 hrs prior to testing. The gauge length was set at 75 mm and the speed at 300 mm/min. Tensile test was carried out in both warp and weft directions.

A Phillips X-PERT PRO X-Ray machine was used to calculate the X-ray crystallinity of the samples. The X-ray source was CuKα radiation (40 kV, 80 mA). Samples were scanned at a scanning rate of 4°/min.

A Shimadzu 8201 Fourier transform infrared (FTIR) spectroscopy was used to find out the reaction between TCA and MC.

A ZEISS (Model: Evo 50) Scanning electron microscopy was used to observe the surface modification of TCAMC treated and untreated fabrics.

2.4. Antimicrobial assessment

The antibacterial activity of untreated and TCAMC-neem oil-treated samples were tested qualitatively by parallel streak method (AATCC-147) and quantitatively by colony counting method (AATCC-100) using Staphylococcus aureus (Gram-positive) and Escherichia coli (Gram-negative) bacteria. In the parallel streak method, bacteria suspension in the form of streaks was placed on the Agar plate with the help of a sterilized wire-loop. Two streaks of 1 cm apart from each other were made on the upper side of the solid Agar in the plate. Thereafter, the fabric swatches (2"×1") of both the untreated and neem oil treated were pressed against the bacterial streak and incubated at 37 °C. After 24 hrs. incubation, the swatches were examined
for any potential bacterial growth underneath and around the specimen. In colony counting method, the swatches were placed separately in a previously sterilized flask containing 10 µl of test organism in Luria broth (Hi-media) solution and kept at 37 °C for 24 hrs in a laboratory shaker at 200 rpm. After 24 hrs incubation, bacteria suspension was diluted serially (like $10^{-3}$, $10^{-4}$, $10^{-5}$ and $10^{-6}$ times) using sterilized water. 10 µl of the diluted bacteria suspension was spread on the Agar plate and the colony growth inside the plates were counted after 24 hrs. incubation at 37 °C [22]. The percent reduction in number of colonies in treated sample as compared to the untreated sample gives the antibacterial activity of the fabric.

Antimicrobial activity or % reduction = $\frac{(A - B)}{A} \times 100$

Where, A is the bacteria colonies (CFU/ml) of untreated fabric and B is the bacteria colonies of the treated fabric.

The neem-oil-treated fabrics were washed in Launder-o-meter according to AATCC test method 124-1975 (II A) using Lissapol N (non-ionic detergent) to check the wash fastness of the treated fabrics.

3. Results and discussion

3.1. Determination of dry add-on% of neem-oil-treated PET

To determine the change in weight of the TCAMC-treated-PET fabrics at different concentrations of TCAMC and also to evaluate the additional weight gain after neem oil application, the dried fabrics were kept at a standard atmospheric
condition of 20 °C and 65 % RH for 24 hrs. before weighing the samples. It is evident from Table 1 that there is no noticeable weight loss due to TCAMC treatment irrespective of concentration. However, an increase in weight due to the introduction of neem molecules into the PET structure is noticed. This supports the findings of the X-ray crystallinity observation (discussed in the later part) that the TCAMC pre-treatment influences the PET polymer structure and the extent of structural change depends on the concentration as evidenced that the increase in concentration enhances the accommodation of more neem molecules into the compact structure of PET (Table 1).

It is obvious from the results that the TCAMC pre-treatment facilitated the entry and entrapment of neem ingredients into the compact polyester structure. As discussed in previous Section, the TCAMC solvent system modified the structure of polyester and created more voids and cracks within the structure to accommodate and entrap the antimicrobial agents present in the neem oil. The increase in concentration of TCAMC treatment facilitated the easy entry and accommodation of substantial amount of antibacterial neem ingredients within the polyester structure which ultimately impart considerable increase in antibacterial activity (discussed in antibacterial activity section).

3.2. Effect of TCAMC on crystallinity of PET fabric
According to the solubility parameter theory it has been established [33, 34] that highly interacting solvents, after diffusion into the polymer structure, promote crystallisation, re-crystallisation of the molecules and increase their segmental mobility so that structural modifications take place. It will be mentioned that PET polymer consists of micro-fibrils that are arranged in columns, and in between these micro-fibrils oriented non-crystalline domains are present. Disordered domains are also present in between the oriented crystallites. These domains are plasticised during polymer-solvent interaction and the solvent-induced crystallisation takes place. The extent of solvent induced crystallisation and consequent structural rearrangement depend on the nature of polymer structure, solvent concentration and the treatment time. It is evident from Table 2 that there is no appreciable increase in X-ray crystallinity at lower concentration of 1% and 3% TCAMC treatment. However, about 6% decrease in crystallinity is observed at 5% TCAMC treated fabric. The changes reveal that the TCAMC treatment influences the crystallinity of PET polymer, thereby indicating structural modification. In other words, solvent induced crystallisation taken place in PET fabric due to TCAMC treatment. In this case the degree of solvent induced modification depends mainly on the concentration of the TCAMC because the treatment time and temperature were kept constant. It will be possible that at higher concentration of TCAMC treatment the PET structure is severely affected resulting higher loss in X-ray crystallinity and tensile strength. As
discussed in preceding section, 25% TCAMC dissolves out the PET polymer in 5min at 30 °C.

It is also observed in Table 2 that the structural modification does not affect the tensile properties noticeably because the change in crystallinity at 5% treated sample due to TCAMC treatment is comparatively low, 6%.

3.3. Observation of surface morphology

Surface morphology of unmodified and TCAMC-treated-PET was studied by using Scanning Electron Microscopy (SEM). It is obvious from Fig. 2 that voids and micro cracks are seen after TCAMC treatment. This reaffirms the earlier discussion that the TCAMC solvent system penetrates into the compact PET structure and creates crakes and pores which facilitate the easy entry of neem molecules.

3.4. Antibacterial activity assessment

3.4.1. Parallel streak method

Antibacterial activity of unmodified polyester, neem-oil-treated unmodified polyester and neem-oil-treated TCAMC modified polyester fabrics were examined qualitatively by parallel streak method and depicted in Fig. 2. Both Gram-positive (Staphylococcus aureus) and Gram-negative (Escherichia coli) bacteria were used for antibacterial assessment.
It is obvious from Fig. 3 that heavy growth of both the *S. aureus* and *E. coli* bacteria was observed in the case of unmodified PET (Fig. 3(a) and (d)) whereas moderate growth was seen in neem-oil-treated unmodified polyester (Fig. 3(b) and (e)). However, it is interesting to note that improved bacterial resistance was recorded in neem-oil-treated TCAMC modified PET in which no substantial bacterial growth was found both on the surface of the fabric and at the fabric edges (Fig. 3(c) and (f)). Visual observation of colour staining between TCAMC modified and the corresponding unmodified ones also qualitatively confirms the degree of antibacterial activity. The enhanced antibacterial efficacy of neem-oil-treated TCAMC modified PET is further reaffirmed quantitatively.

### 3.4.2. Colony count method

The antibacterial activity of unmodified, neem-oil-treated unmodified and neem-oil-treated TCAMC modified polyester was assessed quantitatively against both the Gram-positive and Gram-negative bacteria, and the results are presented in Table 3.

It is evident from Table 3 that neem-oil-treated unmodified polyester fabric registers the antibacterial activity of 16% and 12% against Gram-positive and Gram-negative bacteria respectively. However, there is substantial increase in antibacterial activity of neem-oil-treated TCAMC modified polyester. The antibacterial activity increases with the increase in TCAMC concentration and the increase is more
pronounced at 5% TCAMC pre-treated polyester, 81% and 72% antibacterial activity against Gram-positive and Gram-negative bacteria respectively. It should be noted that the neem oil concentration (10%) is kept constant at various concentration of TCAMC pre-treatment to study the influence of TCAMC on antimicrobial activity of neem-oil-treated polyester. It is obvious from the results that the TCAMC pre-treatment contributed a significant role in enhancing the antibacterial activity of neem-oil-treated polyester. As discussed in the preceding Sections, the TCAMC modified the structure of PET polyester and created more voids and cracks within the structure to accommodate and entrap the antimicrobial agents present in the neem oil. The increase in concentration of TCAMC treatment increases the structural modification and that facilitated the easy entry and accommodation of high amount of antibacterial neem ingredients within the polyester structure and hence the higher antibacterial activity. It should be mentioned that further studies would be carried out to optimise the concentration of TCAMC treatment and time to achieve the highest antibacterial activity without significant decrease in strength loss of polyester fabric. The durability of antibacterial effect on repeated launderings at various concentrations of TCAMC treatment would also be carried out to study the amount of neem antibacterial compounds entrapped within the PET structure, sustained release of neem antibacterial agents from the structure and the degree of antibacterial effect.
3.5. Study of wash durability of the neem oil treated PET

The retention of antibacterial activity of the neem-oil-treated 5% TCAMC modified PET fabrics after 1, 3, and 5 machine washes was studied and the results are depicted in Fig. 4. The antibacterial activity of unwashed but modified and treated fabric was considered as 100% for comparative purpose. It is also considered that one AATCC machine wash is equal to 5 home launderings [22]. It is obvious from Fig. 4 that neem-oil-treated 5% TCAMC modified PET fabric retains substantial antibacterial efficacy against Gram-positive (s. aureus) bacteria even after 3 and at 5 machine wash launderings.

A close look at Fig. 4 indicates that about 80% of antibacterial activity against S. aureus is retained after 1 and 3 washes and the effect is not substantially reduced even after 5 wash (70%). However, the effect is retained by 50% against E.coli after 5 washes, which is equal to 25 home launderings. It is inferred from the wash durability study that the TCAMC modified fabrics not only facilitated the easy entry of neem molecules but also promoted them adhering within the PET structure.

3.6. Kinetic study: bacterial growth

The growth kinetic study of both the S. aureus and E. coli bacteria at different incubation time (2 hrs to 24 hrs) was carried out using 5% TCAMC pre-treated and subsequent 10% neem-oil-treated one washed PET fabrics. The bacterial growth and
antibacterial efficacy of the treated fabrics at different incubation times are depicted in Figs. 5 and 6.

In the case of *S. aureus* (Gram-positive) bacteria, the maximum growth reduction is observed after 2 hrs. of incubation and in other words the antibacterial activity was 100% (Fig. 5(a)). The colonial growth observed in control sample is found to be less as compared to that of longer incubation time and no bacterial colony was found in the treated sample at 2 hrs. incubation period (Fig. 5(b)). With increasing incubation time, the antibacterial activity slowly decreased to 90% and the activity further reduced to 81% when the incubation time is increased to 24hrs. The initial increase in antimicrobial activity after 2hrs of incubation may be explained by the possibility that the interaction of entrapped neem molecules in PET fabric with the bacterial colony is high and this prevents the further propagation of bacteria. On the other hand, as the incubation time increases to 24 hrs. the amount of neem molecules released from the fabric is reduced and consequently further prevention of bacterial growth is reduced. The bacterial growth (CFU) in Agar plates for both the control and neem-oil-treated 5% TCAMC modified PET samples at different incubation time intervals (2, 4, 6, 24 hrs.) is shown in Fig. 5(c).

In the case of *E. coli* (Gram-negative) bacteria, 100% antibacterial activity is observed after 2 hrs. of incubation (Fig. 6(a)). However, as the incubation time increases the antibacterial efficacy of the treated sample gradually decreased (Fig. 6(c)) and finally reached to 72% after 24 hrs. of incubation. The reduced antibacterial
activity at higher incubation time is perhaps, as discussed above in the case of *S. aureus*, due to the reduced amount of release of entrapped neem molecules from the treated PET fabric. From the Figs. 5 and 6, it can also be concluded that the neem-oil-treated sample showed higher antibacterial activity against Gram-positive *S. aureus* than that of Gram-negative *E. Coli* irrespective of incubation periods. The greater resistive power of *E. coli* bacteria (Gram-negative) may be due to the presence of an additional outer membrane made up of lipopolysaccharide and protein which is absent in Gram-positive (*S. aureus*) bacteria [35, 36].

4. Conclusions

The solvent induced crystallisation takes place in PET polymer after pre-treatment with TCAMC reagent. The pre-treatment modified the structure of PET polyester fabric and thereby created more voids/cracks within the structure. The degree of structural modification depends on the concentration of TCAMC reagent. The changes in X-ray crystallinity indicate the extent of the structural modification of PET affected by TCAMC. The large increase in crystallinity is more pronounced at lower concentration of 1% TCAMC and thereafter the effect is decreased at higher concentrations of 3% and 5%. The pre-treatment does not alter the tensile properties noticeably even at the maximum concentration of 5% for 5 min treatment. The pre-treatment facilitated the easy entry of antibacterial compounds present in the neem oil and consequently this enhanced the antibacterial activity of the polyester fabric.
Higher antibacterial activity against both the Gram-positive and Gram-negative bacteria is observed at 5% TCAMC pre-treated and neem-oil-treated polyester fabric. The durability of antibacterial effect against both the Gram-positive and Gram-negative bacteria of TCAMC pre-treated and subsequent neem-oil-treated PET fabrics is substantial up to five machine washes which is equivalent to 25 home launderings. It is demonstrated that the neem molecules are effective antimicrobial agent against both the Gram-positive and Gram-negative bacteria and the efficacy of antimicrobial activity of treated polyester fabric depends on the amount of neem molecule released from the PET structure. The TCAMC treatment facilitated the easy entry of neem molecules into the compact structure of PET and also enhanced the accommodation of the molecules within the structure.

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References

Table 1: Change in weight of TCAMC and neem oil treated PET fabric.

<table>
<thead>
<tr>
<th>TCAMC (%)</th>
<th>Initial fabric weight (gm)</th>
<th>Weight after TCAMC treatment (gm)</th>
<th>Weight change (∆)</th>
<th>Weight after neem oil treatment (gm)</th>
<th>Add-on (%)</th>
<th>CV % of Add-on</th>
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Table 2: Crystallinity and tensile properties of TCAMC treated PET

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<th>TCAMC % (w/v)</th>
<th>X-ray crystallinity (%)</th>
<th>Load at break (kg)</th>
<th>CV of breaking load (%)</th>
<th>Displacement at Break (mm)</th>
<th>CV of breaking displacement (%)</th>
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<tr>
<td>Description</td>
<td>Neem oil (%)</td>
<td>S. aureus (Gram-positive) CFU/ml $\times 10^7$</td>
<td>Antibacterial activity (%)</td>
<td>E. coli (Gram-negative) CFU/ml $\times 10^7$</td>
<td>Antibacterial activity (%)</td>
</tr>
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<td>---------------------------------------------</td>
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<td>Unmodified polyester</td>
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<td>81</td>
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Fig. 1(a)
Fig. 1(b)
Fig. 1(c)
Fig. (2)

a. Untreated PET fabric  
b. 5% TCAAMC treated PET fabric
Fig. (3)
Fig. (4)
Fig. (5)
Fig. (6)
Figure Captions

Fig. 1(a). FTIR spectrum of 100% trichloroacetic acid (TCA)

Fig. 1(b). FTIR spectrum of 100% methylene chloride (MC)

Fig. 1(c). FTIR spectrum of 5% trichloroacetic acid and methylene chloride (TCAMC)

Fig. 2. SEM images of (a) unmodified (b) 5% TCAMC treated PET.

Fig. 3. Antibacterial effect against S. aureus bacteria (a-c): (a) unmodified polyester (b) neemoiltreated unmodified polyester (c) neem oil treated 5% TCAMC modified polyester; and against E. coli bacteria (d-f): (d) unmodified polyester (e) neem oil treated unmodified polyester (f) neem oil treated 5% TCAMC modified polyester.

Fig. 4. Durability of antimicrobial activity after several machine washes.

Fig. 5. Growth kinetic study of S. aureus bacteria with neem oil treated TCAMC modified washed sample at different incubation time (A) antibacterial activity (B) growth of colony forming unit / ml and (C) picture of colony forming unit in Agar plate, for both control & treated sample.

Fig. 6. Growth kinetic study of E. coli bacteria with neem oil treated TCAMC modified washed sample at different incubation time (A) antibacterial activity (B) growth of colony forming unit / ml and (C) picture of colony forming unit in Agar plate, for both control & treated sample.