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Research Article

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Antibacterial finishing application of N-phthalimido-α-hydroxyaceticacid

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ABSTRACT

The antimicrobial finishing of textiles is intended to provide protection of textiles against microbial corrosion, prevention of malodor or prophylaxis and therapy of infections, respectively. This article evaluates the applicability of N-Phthalimido-α-hydroxyaceticacid as antibacterial in textile finishing. It was synthesized by the reaction between Phthalimide and Glyoxalic acid in THF medium. The synthesized compound was identified by spectral *data and covalently attached to the cotton fabric* by a two-stage process of chemical modification. *The existence of chemical linkage was confirmed by SEM analysis and F.T.I.R. The treated fabric was subjected to multiple washings to determine durability. The treated fabric was then analysed for antibacterial properties.*

Key words: antimicrobial finishing, N-Phthalimido- α-hydroxy-aceticacid, cotton fabric, covalent linkage, durability, antibacterial activity.

INTRODUCTION

Recent market survey has quite convincingly shown that the apparel consumers all over the world are demanding functionality in the product. Anti microbial textiles with improved durability find a variety of applications such as health and hygiene products, specially the garments worn close to the skin and several medical applications, such as infection control and barrier control. Therefore antimicrobial finishing[1-4] is increasingly present in the literature due to the high interest in the market.This work looked at the feasibility of utilizing a new antibacterial drug N-Phthalimido-α-hydroxyaceticacid[5] and chemically converting it in order to obtain a reactive dye type molecule, which could be applied to cotton fabric with the goal of imparting the antibacterial properties of the antibiotic compounds to the fabric.

EXPERIMENTAL SECTION

Melting point was determined by open ended capillary tube in the electrical melting point apparatus and are uncorrected and the purity of the compound was checked by TLC. The structure of the compound was confirmed by IR (Perkin-Elmer) in KBr disc and 1H NMR (Bruker Spectrospin AV 400 MHz Spectrometer using TMS as an internal standard).

Test fabric:
The fabric was white, scoured and bleached 100% cotton, tight-weave fabric, weighing approximately 140g/m².

Synthesis of N-Phthalimido-α-hydroxy acetic acid (NPHA):
Phthalimide 10 g (68 mmol) was dissolved in THF (200 cm^3) at room temperature with stirring. To this was added glyoxalic acid (50% wt) 30.2 g (0.204 mol) and the reaction mixture was heated to reflux for 3Hrs. Evaporation to dryness under reduced pressure produced a cream solid. This was recrystallised from ethyl acetate to give a white solid (13.19g, 88%). Melting point 191^oC.

Conversion of NPHA in to its potassium salt:

11 g (0.05 mol) of NPHA was dissolved in 60 cm^3 of chloroform. The resultant solution was then neutralised with 2.8 g (0.05) mol of KOH dissolved in 50 cm^3 of ethyl alcohol. The potassium salt of NPHA was precipitated by pouring the mixture into dry acetone. After filtration, the salts were dried under reduced pressure at 50 ^oC to a constant weight .

Antimicrobial finish application:

N-Phthalimido-α-hydroxy acetic acid was applied to the cotton fabric by covalent bonding method[6]. This method involves the following two steps.

STEP-I: Reaction between cotton fabric and chloroacetyl chloride

5 x 5 cm cotton fabric (1.4 g) was placed in 250 cm^3 round-bottom flasks equipped with a stirrer, and then 60 cm^3 THF and 1.0 cm^3 (13.4 mmol) pyridine were added. The mixture was cooled to 0^oC.Then 1.0 cm^3 (12.4 mmol) chloroacetyl chloride dissolved in 5 cm^3 THF was added in a drop wise manner. The reactions were carried out at 25 ^oC in a nitrogen atmosphere. After 24 h, the fabric samples were separated from the precipitated pyridine hydrochloride. The fabric was carefully washed with water and then with ethanol to remove impurities. Finally the fabric was dried under reduced pressure at 40^oC to a constant weight.

STEP-II: Reaction between chloroacetylated cotton fabric and potassium salt of NPHA

The chloroacetylated cotton fabric (5×5 cm) was placed in 250 cm^3 round-bottom flask equipped with a magnetic stirrer, and 50 cm^3 of DMSO was added. A solution of 1.2 g (18 mmol) of potassium salt of NPHA dissolved in 5 cm^3 DMSO was added. The reaction was carried out at 30^oC with intense stirring for about 5 h. Next, the fabric sample was carefully washed with ethyl alcohol to remove un reacted potassium salt of NPHA, and then dried under reduced pressure at 60^oC to a constant weight.

Testing and analysis:

SEM and FTIR analysis:

Surface morphological structure of untreated and treated fabric was measured with a SM-5600LV scanning electron microscopy (SEM). Fourier transform infrared spectroscopy (FTIR) was employed to study the linkage between the cotton fabric and NPHA.

Antimicrobial activity assessment:

Antimicrobial activity was evaluated by both qualitative and quantitative test methods (AATCC-147, 2004).

Qualitative analysis:

Anti microbial activity testing[7-10] of the NPHA treated fabric was carried out qualitatively by agar diffusion method. In this method the treated and an untreated control fabric were pressed into intimate contact with Müller-Hinton agar medium inoculated with Escherichia coli (ATCC 15223). If antibacterial activity is present, it will be possible observe a clear zone around the treated sample comparing to the zone of bacterial growth around and over the untreated control sample after the same contact time. These qualitative methods provide a formula to measure the inhibition zone width, but this is a qualitative evaluation and it cannot be considered as a quantitative indication of the antibacterial activity.

Quantitative analysis:

Quantitative antimicrobial testing was carried out by using the shake flask method. Tests were conducted against the *Staphylococcus aureus* ATCC 6538 Artificial concentration of bacterium that is 2.8 x 104 /ml was treated with 1 g of the fabric separately in a test tube. The test tube was shaken at 35ºC for 1 h on a rotary shake at 100 rpm. The reduction of colonies was calculated using the following equation:

$$\text{Reduction rate in the number of colonies (\%)} = \frac{(A-B)}{A} \times 100$$

Where;

A = Number of colonies before shaking.

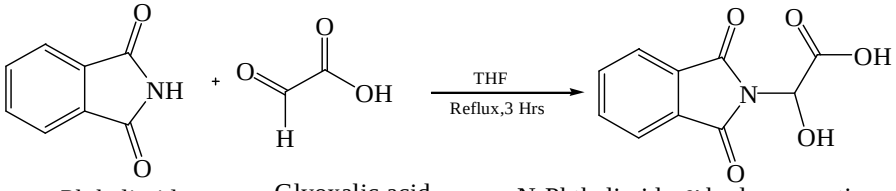
B = Number of colonies after 1 hour shaking.

Durability Testing:

To test the durability [11-15] of the antimicrobial finish, treated samples were examined for antimicrobial efficacy after 3, 5, 10, and 20 home launderings. The procedure used for home laundering was the AATCC Test Method 61. The detergent used for this test was the AATCC standard reference detergent.

RESULTS AND DISCUSSION

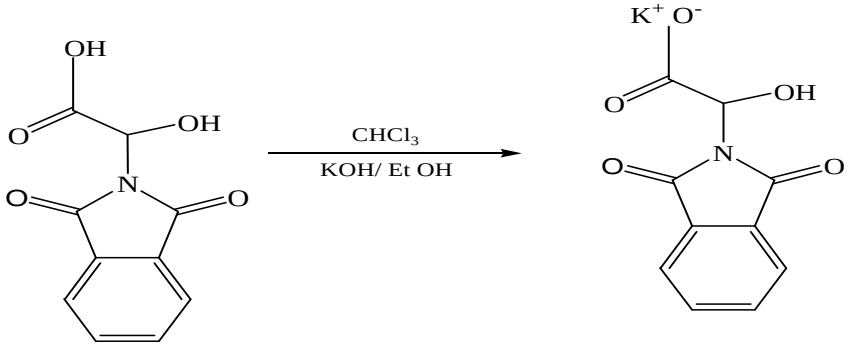
Synthesis of the target compound **(3)** was achieved by reacting phthalimide **(1)** with glyoxalic acid **(2)** in THF medium **(Scheme I)**.



Phthalimide **1** Glyoxalic acid **2** N-Phthalimido-α-hydroxy acetic acid (NPHA) **3**

Scheme I

The potassium salt **(4)** was obtained by treating **(3)** with alcoholic potassium hydroxide **(Scheme II)**.



3 **4**

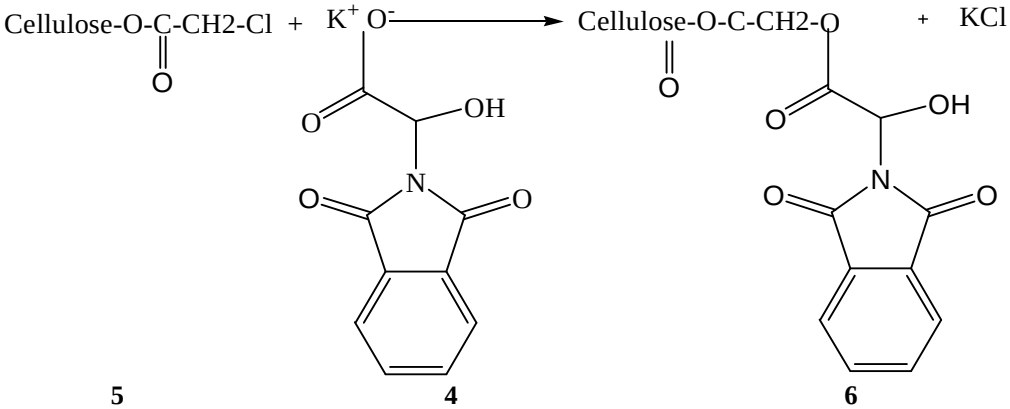
Scheme II

Chloroacetylated cotton fabric **(5)** was obtained by the reaction between cotton fabric and chloroacetylchloride **(Scheme III)**.

5

Scheme III

The reaction between chloroacetylated cotton fabric **(5)** and potassium salt of NPHA **(4)** yielded NPHA incorporated cotton fabric **(6) (Scheme IV)**.



5 **4** **6**

Scheme IV

Characterisation of NPHA: The formation of product NPHA was confirmed by FTIR and proton NMR.

Table.1 Spectral data of NPHA

IR (KBr, Cm^{-1})	^1H NMR (CD_3OD, ppm)
3458-3058(Ar),1745(-C=O), 740-646, 1502-1303 (O-H bending),3625 (O-H stretching),1303(C-N stretching)	7.8(s, Ar-H), 11.3(m, -COOH), 5(m, Al-H), 2(s,-OH)

SEM analysis

The surface characteristic and the structure change of treated cotton fabric was investigated by means of scanning electron microscopy, surface morphological structure of untreated ,chloro acetylated and NPHA incorporated cotton fabrics were shown in Fig.1 and Fig.2. and Fig. 3

As can be seen, the surface of the elementary cotton fibres of the unmodified fabric (Fig 1) is smoother and more homogeneous than that of the chemically modified fabric (Fig 2 and 3). The surfaces of both modified fibres are rougher.

SEM photographs (900 x)

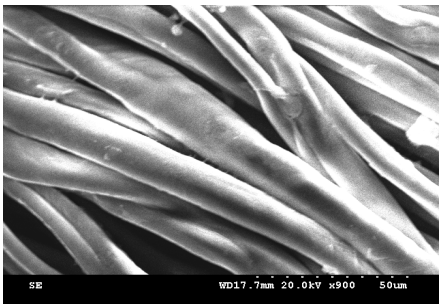


Fig 1 Un treated cotton fabric

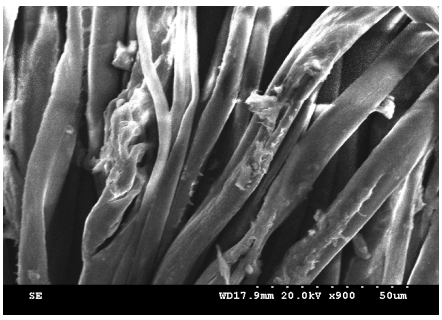
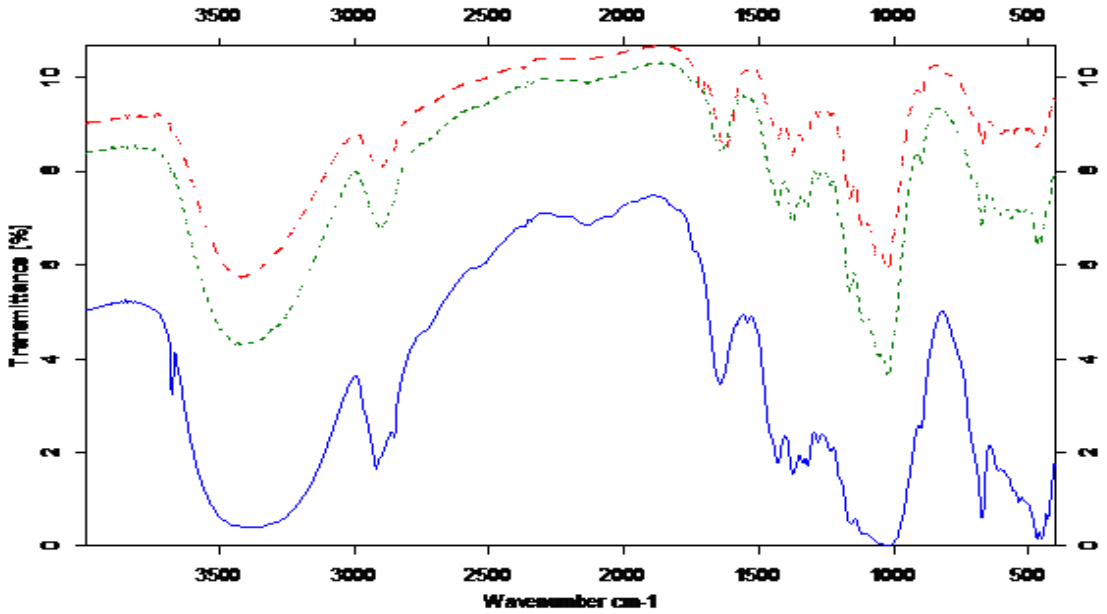


Fig 2 Chloro acetylated cotton fabric

Fig 3 Cotton fabric containing incorporated NPHA

FTIR analysis

FTIR spectrum of untreated, chloro acetylated and NPHA incorporated cotton fabrics were shown in Fig 4.



Untreated cotton fabric
Chloro acetylated cotton fabric
NPHA incorporated cotton fabric

Fig 4 FTIR spectra

Unlike the spectrum of the unmodified cotton fabric, the spectrum of the chloroacetylated fabric shows a new weak signal at 1764 cm-1 derived from the ester groups >C=O, while the NPHA incorporated cotton fabric reveals a higher intensity of the band of ester groups at 1713 cm-1 as well as a band at 785 cm-1, which results from scissoring vibration bands >C=C< and C-H in the Phthalimide ring. Hence an analysis of the results of FTIR spectroscopy confirmed the existence of the chemical linkage between cellulose chains and NPHA.

Antibacterial activity assessment :
Qualitative analysis:

Table 2. Antibacterial assessment by agar diffusion method

Sample No	Nature of Fabric	No of wash cycles	Width of the inhibition zone (mm)
1	Untreated	-	0
2	Treated	0	12
3	Treated	5	11
4	Treated	10	10
5	Treated	20	8
6	Treated	40	6

Quantitative analysis:

Table 3. Antibacterial assessment by shake flask method.

Sample No	Nature of Fabric	No of wash cycles	Bacterial reduction (%)
1	Untreated	-	0
2	Treated	0	99.8
3	Treated	5	99.1
4	Treated	10	98.7
5	Treated	20	98
6	Treated	40	97.6

It can be seen from the test results in Table 3 that the antimicrobial activity decreased with increasing number of washings. The treated fabrics showed good wash fastness as expected.

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CONCLUSION

In conclusion, a simple method has been developed to prepare N-Phthalimido-α-hydroxyaceticacid (NPHA) and the incorporation of the same on cotton fabrics to impart antimicrobial properties. The test results are showed that the NPHA treated fabric showed excellent antimicrobial activity.

Wash durability is also good. Durability to washing of NPHA treated cotton fabric is 97.6% after 40 washing. From this study it can be suggested that the NPHA treated fabrics can be used for textile application.

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