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Investigation into the Effects of an Easy Care Cross-linking Agent on the Properties of the Pulp Reclaimed from Cotton Based Waste Garments

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Author's contribution

The sole author designed, analyzed and interpreted and prepared the manuscript.

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Original Research Article

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ABSTRACT

Aims: To investigate the potential pulp reclamation from easy care treated cotton waste garments for potential paper making and regeneration of fibres.
Study Design: This is an experimental laboratory research project.
Place and Duration of Study: School of Materials, University of Manchester, between October 2009 and March 2013.
Methodology: Cotton fabric was treated with Dimethylol dihydroxyethylene urea (DMDHEU) at 60, 100 and 140 g/L concentration levels. The fabrics were then shredded and pulped on laboratory Valley beater. Part of the resultant pulp was formed into hand sheets using the sheet making apparatus. The remaining pulp was analyzed for the wetness test, fines content and fibre length analysis. Tensile strength and Tear strength of the hand sheets were analyzed using Instron tester and Elmendorf machines, respectively. The Kawabata Evaluation System for Fabrics was used to assess the hand properties of the hand sheets. X-ray photoelectron spectroscopy technique was used for the surface chemical analysis of the hand sheets.
Results: It was observed that as the concentration of DMDHEU reacted to cellulose polymer increased, shorter fibres were produced. The DMDHEU free pulp has a mean fibre length of

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2.5±0.04 mm whereas after increasing the DMDHEU concentration equivalent to 1.6% surface nitrogen the mean fibre length dropped to 1.2±0.05 mm. Application of the DMDHEU easy care finish on the cotton fabric reduced the tensile strength of the hand sheets from 2.08 kN/m to 0.1 kN/m whereas the tear strength and tensile index were reduced from 1.4 kN to 0.5 kN and 0.9 KNm/g to 0.4 kNm/g, respectively.

Conclusions: The DMDHEU easy care finish produced pulp with shorter fibres, high fines content but hand sheets with relatively weaker mechanical properties. This implies that in order to recover fibres from the cotton waste garments for either fibre regeneration or paper processing the DMDHEU easy care finish must be removed prior to forming of hand sheets.

Keywords: Cotton; waste garment; easy care; beating; DMDHEU.

1. INTRODUCTION

Most natural and synthetic fabrics garments can be recycled. The original collection of used textiles and clothing was done by the 'rag and bone men', a practise that started in the Yorkshire Dales, United Kingdom, some 200 years ago [1]. The collected garments were broken down into their constituent fibres (shoddy), so that they could then be re-spun into new yarns [2].

Before the emergence of the synthetic fibres, cotton rags were used as raw material for paper making. However following the emergence of the former and advancement in fibre blending technology, the separation of synthetic fibre components from the fabrics has become more difficult and this may be considered as a major reason for the decline in the use of cotton rags in paper making. The fast change in fashions and lower prices of clothing has contributed to the increased volume of textiles consumed [3,4]. These used clothes can be disposed off in various ways and the most common route is export to developing countries and sold as second hand clothes [3,5]. The decline in production, closure of businesses and failing of the textile industry in developing countries can be attributed to amongst other factors the second hand clothing imports [6,7]. The second hand clothes business in sub-Saharan Africa countries is not well regulated and this has resulted in custom frauds whereby traders have tried to avoid taxes [6]. As a response to this trade, 14 countries in Africa, as well as some countries in Latin America and Caribbean, are banning imports of recycled clothing or making it bureaucratically impossible to import [8]. The arguments that second hand clothing businesses provide employment and income to developing economies [6,9] cannot be a support for the continuing use of second hand garments instead of getting involved in the production of new garments. The second hand clothes markets in Africa are declining due to the increase in cheap imports from Asia [10]. This has resulted in increased land filling costs at the origin of the second hand clothing [4]. Thus it is necessary to explore more methods of recycling waste garments for sustainability of the global textile and apparel industry [11].

There are obvious economic and environmental reasons for textile recycling and these include: reduction of pollution created during incineration processing; efficient use of landfill space; diversification of applications of textile material through conversion of textile wastes into products other than clothing; and to provide clothing at reasonably low cost to underprivileged countries [12,13]. However, the supply of second hand clothing to developing countries as a reason for helping the poor [13] is a threat to the destination textile and garment industry [7,8].

Prior to export to developing countries textiles materials from consumers reach recycling centres through private jumble sales, charity shops, textile banks, door to door collection, kerbside recycling schemes and direct disposal [3]. After collection, the waste materials are taken for sorting into various grades [14]. Typically sorting is performed manually, making the process labour intensive and some automatic sorting technologies have been reported [15,16]. The high labour costs in developed countries encourage shipment of unsorted used clothing to developing countries, however the option is not sustainable due to healthy and safety issues during sorting at the destination countries [5]. These restrictions necessitate not only advanced sorting technology but also alternative methods of recycling of used garments other than export to developing countries for re-use.

The commonest method for disintegration of the waste garments for recycling is by mechanical shredding [3,17-22]. During shredding, the garment is subjected to a rotating drum equipped with wires while held with nip rollers; the pins on the surface of the rotating drum disintegrate the fabric into yarns and subsequently into fibres. Reclaimed fibres are carded and then made into new products, mainly nonwoven products. Due to the quality of the fibre reclaimed from the shredding process, conventional spinning results in yarns with low twist and relatively weak mechanical properties. Thus nonwovens are the most common market for recycled fibres. Yarn spun from 100% recovered fibres with acceptable commercial qualities can only be produced by pneumatic spinning processes [22], but this process needs further improvement due to high breakage. Mixtures of virgin fibres and reclaimed fibres to make new yarns could work for pneumatic spinning whereby yarn of 80-250 tex have been achieved by a series of cards [22]. Thus with the current limitations of the conventional recycling approaches, chemical recycling of PET from both waste bottles and garments [23] and cellulose from waste cotton garments [24,25] have been explored.

The use of waste cotton garments for regeneration of new fibres has been patented and reported [26]. During the fibre regeneration process, the waste garments are cleaned and converted into pulp and subsequently dissolved in amine oxide solution prior to extrusion into fibres. In the previous study there were no considerations about the effect of finishes such as DMDHEU on the quality of the reclaimed pulp. In this paper an investigation on the effects of an easy care finish on the properties of pulp reclaimed from DMDHEU treated cotton fabrics was conducted. The fabrics were deconstructed into pulp by a beating process using a Valley beater. The effects of the DMDHEU on physical properties of the resultant pulp were investigated in order to assess potential pulp making and subsequent cellulose dissolution for fibre regeneration.

Traditionally beating is the mechanical mixing of pulp in water so that the suspension acquires properties that will determine the properties of the ultimate product. During the beating process, the stock is allowed to flow between the rotating beater roll and stationary bedplate. Fibres are mechanically broken by the shearing and friction forces between the fibre and beater roll bars and bed plate bars [26,27]. The objectives and principles of beating are discussed in more detail in the cited references [27-32].

2. METHODOLOGY

2.1 Materials

A plain woven, scoured, bleached, 100% cotton fabric was used throughout this study and was donated by Lenzing, Grimsby UK. DMDHEU (Fixapret CP new) was supplied by Dystar and magnesium chloride was purchased from Fisher Scientific, UK.

2.2 Application of DMDHEU

The cotton fabrics were padded at 80% wet pick up (w.p.u.) with solutions containing 60, 100 and 140 g/L of DMDHEU with an associated magnesium chloride catalyst concentration of 12, 20 and 28 g/L, respectively. The padded fabrics were oven dried for 2.5 minutes at 100℃ with subsequent curing for 4 minutes at 150℃. The cured fabric was rinsed in warm water and then air dried prior to further analysis.

2.3 X-ray Photoelectron Spectroscopy (XPS) Analysis

XPS analysis was performed using a Kratos Axis system spectrometer. The fabric samples were cut from the middle of the specimen and attached to the sample holder using a double sided tape. Monochromatic Al Kα X-rays (1486.69eV) with a power of 150W was used to irradiate the samples. A wide scan spectrum was recorded with pass energy of 160eV from which the surface composition (C, O and N) was determined. A high resolution Nitrogen (1s) spectrum was recorded with pass energy of 40eV. The binding energy (BE) values were calculated relative to the Carbon (1s) photoelectron at 285.0eV. Charge compensation for the samples was achieved using a 4-7 eV beam at a flood current of approximately 0.1mA, with an electrically ground 90% transmission nickel mesh screen. Data analysis was performed using the CASA XPS software [33].

2.4 Laboratory Beating

The DMDHEU treated and DMDHEU free samples were cut into pieces of 10mm x 10mm and fed into laboratory Valley beating machine (E633816). The machine and stock parameter were set in accordance to the TAPPI standards for beating - TAPPI T 200 sp-96. The beating process was carried out for 90 minutes.

2.5 Forming of Hand Sheet for Physical Tests

The hand sheets were made by pulp evaluation apparatus (Mavis Engineering Company) as per TAPPI standards for forming of hand sheets - TAPPI T205 sp-95 and then air dried for 24hours prior to further analysis. At least 14 sheets were made for each DMDHEU concentration level.

2.6 Pulp Wetness Test Analysis

The Schopper-Riegler wetness test was carried out by using Leverantor AB Lorentzen apparatus in accordance to British Standard test method- BS 6035: Part 1. At least five replicates for each DMDHEU concentration level were performed and the mean reported.

2.7 Fibre Length Analysis

The fibre length of the pulp was analysed by using Projectina projection microscope. The microscope comprised a light source condenser, X-Y stage, objective, eyepiece, scaled circular screen and computer. The test fibres were conditioned at 20℃ and 65% relative humidity for 24 hours before testing and then were spread separately on a slide of 75mm x 40mm, mounted with liquid paraffin and finally covered with a 50 mm x 35 mm cover slip. The magnified projected images of fibres were captured on the camera and passed to the computer, and then the length of each fibre was recorded following pencil tracing supported by PIA 400 digital software. In order to ensure accurate measurements random samples were presented on the slide and were measured following the recommended procedures [34].

2.8 Fines Content

The fines content of the stocks were determined by dynamic drainage jar, whereby a single screen classifier was used. The fines content was determined in accordance to TAPPI standard test method - TAPPI T 261 cm-94. Three replicates were performed for each DMDHEU concentration level and the mean reported.

2.9 Mechanical Properties of Hand Sheets

An Instron Series IX instrument was used to determine the tensile strength of the hand sheets. The tensile tests were set according to British Standard test method - BS EN ISO 1924:1995. The machine was set at crosshead speed of 10 mm/min and full scale load range of 0.5 kN.

An Elmendorf machine was used to determine the tear strength of the hand sheets. The tests were in accordance to the British standard test method - BS EN 21974: 1994. The tensile index of the hand sheets were determined by the use of Zero strength machine. Ten replicates were performed per each mechanical test from each DMDHEU concentration level and the mean reported. Prior to testing for mechanical properties the sheets were conditioned at 50% relative humidity and 23℃ for 24 hours.

2.10 Kawabata Evaluation System for Fabrics (KES-F)

With the exception to the tensile, the bending, shear, surface and compression analysers were utilised for KES-F analysis [35-37]. The test conditions for the hand sheets were at 0.8° for both bending (B) and shear rigidity (G). The test conditions for compression properties were 50 g/cm^2 for both compression energy (WC) and thickness (Tm) whereas the test condition for the thickness (To) was $0.5g/cm^2$. The sheets were conditioned at 65% relative humidity and 20℃ for 24 hours prior to testing. Three replicates were performed per sample per test and the mean reported.

3. RESULTS AND DISCUSSION

3.1 The Effect of DMDHEU Concentration on the Fibre Length and Wetness of the Reclaimed Cotton Pulp

The amount of the DMDHEU which has chemically reacted with the cellulose polymer was measured by the XPS analysis of nitrogen assigned to the DMDHEU. While the wide scan analysis of the XPS spectra of the cotton fabrics treated with increasing concentration of DMDHEU revealed an increase in the N(1s) atomic composition with DMDHEU concentration, the N(1s)high resolution spectra indicated an increase in the peak intensity, Figs. 1, 2 and 3. This indicates an increase in the amount of DMDHEU bound to the cellulose polymer with increase in DMDHEU concentration in the padding solution.

Since the surface nitrogen content is a "good" indicator of the amount of DMDHEU bonded to the cellulose chains, therefore all other pulp and hand sheet properties are presented and discussed with the respect to the change in XP surface nitrogen atomic composition.

The effects of DMDHEU on the properties of the reclaimed pulp were investigated in terms of fibre length, pulp wetness and mechanical and hand properties of the hand sheets made from the reclaimed pulp. Examination of the mean fibre length of the pulp reclaimed from the easy care finished cotton fabrics indicated that as the concentration of DMDHEU reacted to cellulose polymer increased, more and more short fibres were produced. For example the DMDHEU free pulp has a mean fibre length of 2.5±0.04 mm whereas after increasing the DMDHEU concentration equivalent to 1.6% surface nitrogen the mean length of the fibre dropped to 1.2±0.05 mm, Fig. 4. The increase in the reduction of fibre length with the amount of DMDHEU bonded to the cotton material is due to the increased brittleness of the fibres of the easy care treated fabrics and the subsequent deconstruction processing breaking up the fabric into shorter fibres. The brittleness is due to the presence of strong crosslinks introduced into the cellulose polymers which when force is applied the fibre deforms inelastically.

The effect of fibre embrittlement caused by the easy care finish was also observed in the pulp wetness properties, Fig. 5, where the effects of surface nitrogen content, assigned to chemically bound DMDHEU (measured by XPS), was related to the pulp wetness and fines contents.

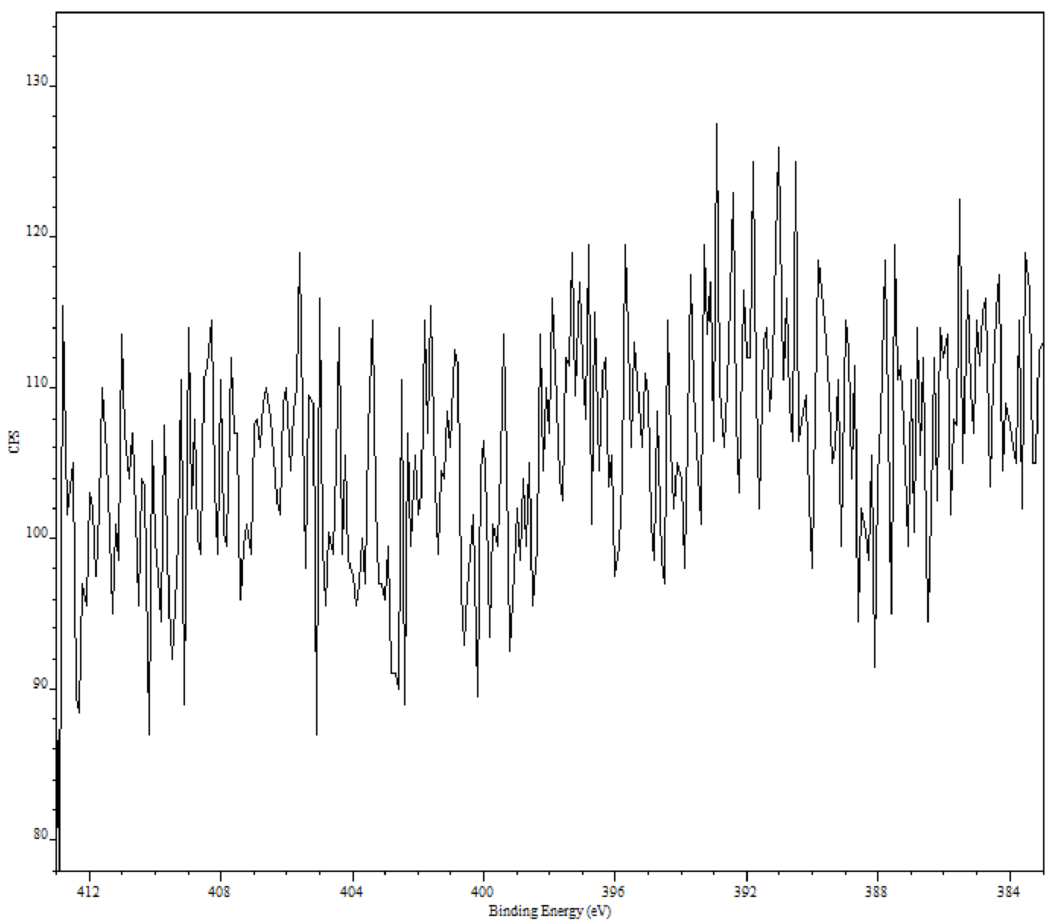

Fig. 1. XP N(1s) spectrum of DMDHEU free cotton fabric

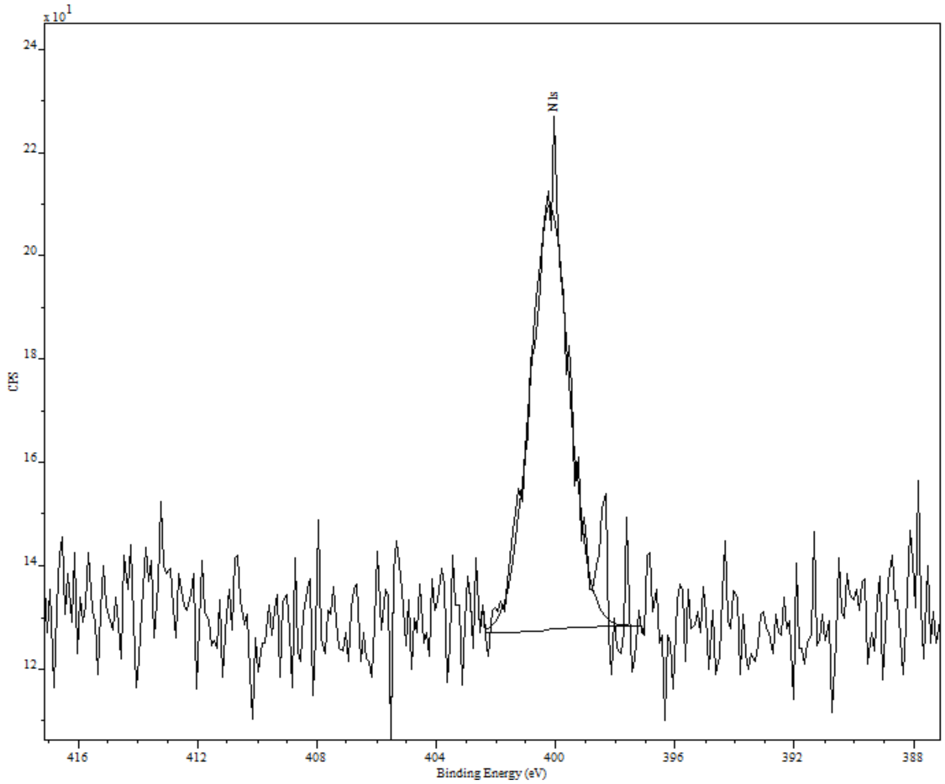

Fig. 2. XP (N1s) spectrum of sheet made of waste cotton treated with 140 g/L DMDHEU

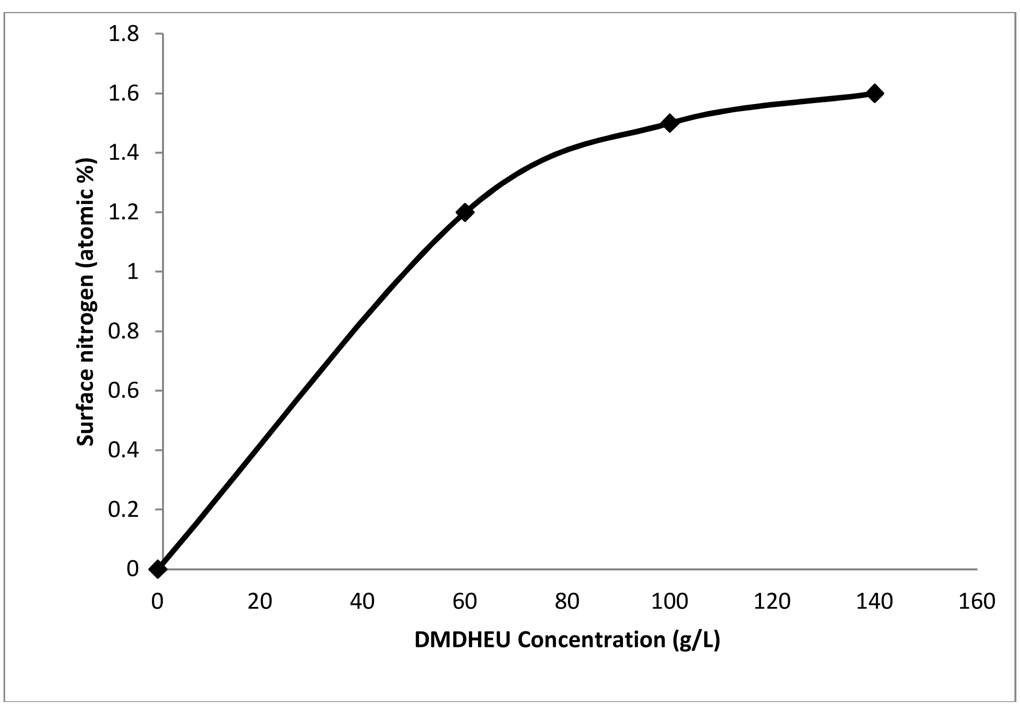

Fig. 3. Variation of XP surface nitrogen atomic %, assigned to DMDHEU, of cotton fabric treated with DMDHEU at increasing concentration

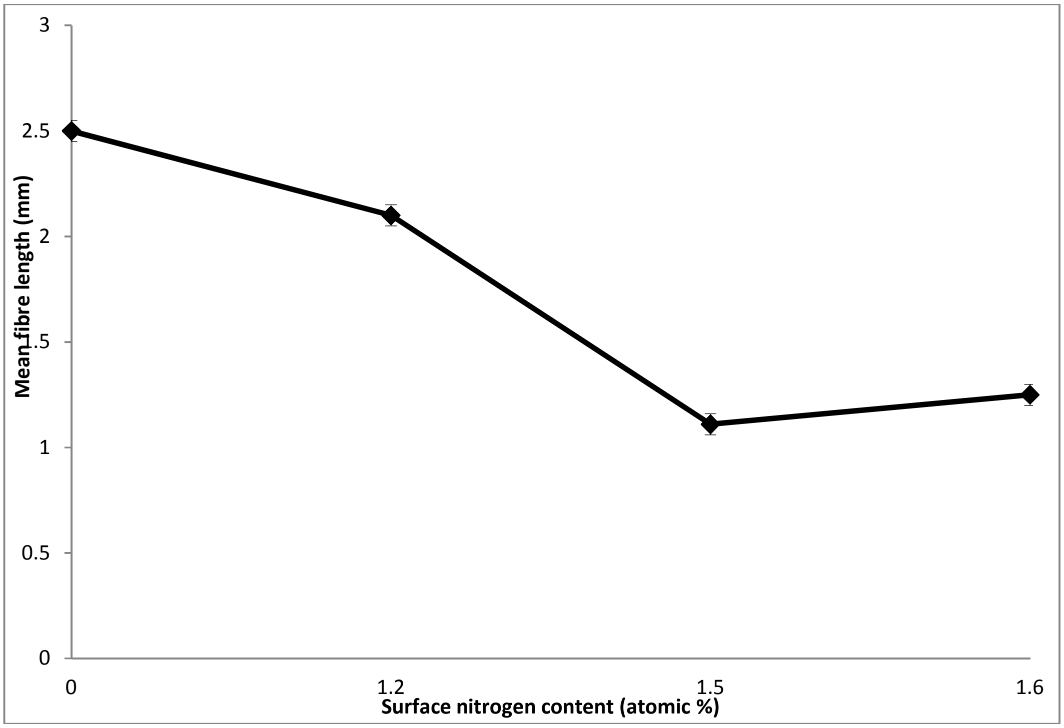

Fig. 4. Variation of mean fibre length with surface nitrogen assigned to reacted DMDHEU

Pulp wetness is the ability of pulp to resist water drainage. Fines content is the fraction of pulp particles that can pass through a round hole, 76µm in diameter, in a 200 mesh screen. Examination of Fig. 5 indicated that both Schopper-Riegler values and fines content of the reclaimed pulp increased as the surface nitrogen assigned to the chemically bound DMDHEU increased. The increase in the Schopper-Riegler wetness with the surface nitrogen assigned to DMDHEU is not unusual. Apparently the ability of the pulp to resist water drainage is due to an increase in fines that are rich in hydroxyl groups exposed on the surface. When the pulp is in water suspension, the surface hydroxyl groups of the fibres will form hydrogen bonds with the water. Thus the resistance to drainage by the pulp is partly due to the water forming hydrogen bonds with the surface hydroxyl groups of the pulp and partly due to physical barrier caused by the fines contained in the pulp. In this case, it is presumed that the physical blockage of the fines is the dominant factor because the DMDHEU crosslinks has reduced the ability of the fibres to form hydrogen bonds with water and is explained in the subsequent subsection. Accordingly the increase in wetness and fines content of the pulp is explained by the increase in shorter fibres in the pulp which is caused by the effect of DMDHEU on fibre brittleness.

3.2 The Effects of the DMDHEU on the Mechanical Properties of the Hand Sheets Made from the Reclaimed Pulp

The tensile strength, tear strength and tensile index of the hand sheets, Fig. 6, indicated that an increase in the amount of DMDHEU reacted to the cotton hand sheets resulted in a loss in strength of the hand sheets.

The loss in strength of the hand sheets with the increase in DMDHEU reacted was attributed by two factors. First is the effect of fibre length on the strength of hand sheet. Since the fibre length is getting shorter as the DMDHEU concentration increased, these shortened fibres in the sheet will act as a series of weak points in which under relatively low load those points will be susceptible to failure. The second factor could be the effect of DMDHEU crosslinks on the bond formation among the fibres in the sheet. It is known that the DMDHEU crosslink by forming ether bonds with hydroxyl groups of the cellulose polymer. The crosslinks can be either bi-, tri- or tetra-functional depending on the concentration level of the applied DMDHEU [38].

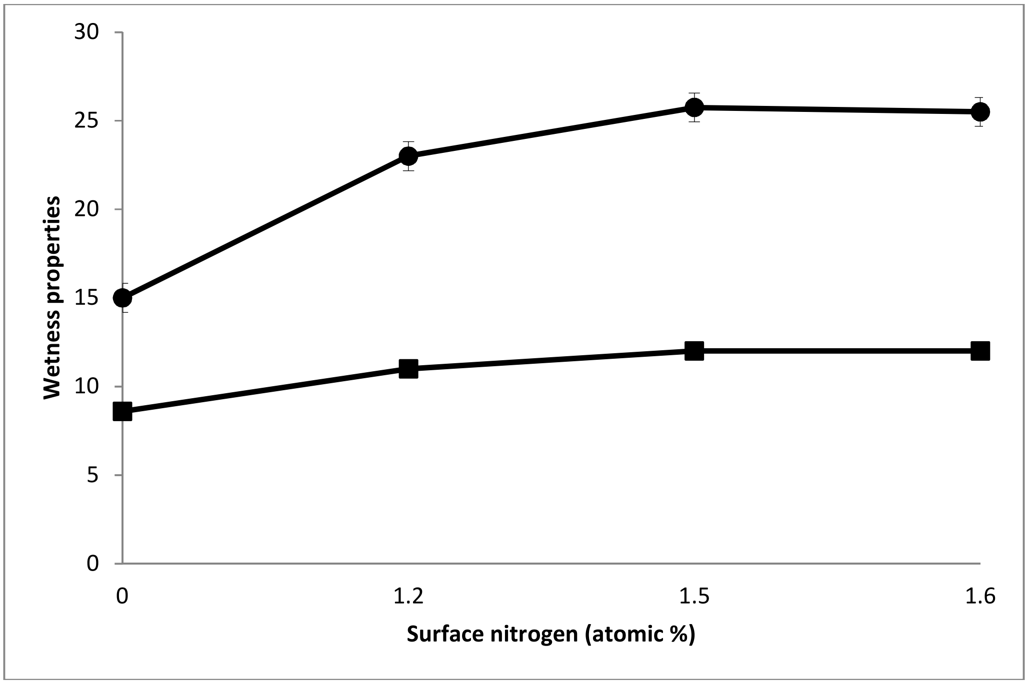

Fig. 5. Variation of surface nitrogen assigned to DMDHEU with wetness properties of cotton pulp reclaimed from cotton fabrics treated with increasing concentration of DMDHEU
●- *Schopper-Riegler (°SR) and* ■-*fines content (%)*

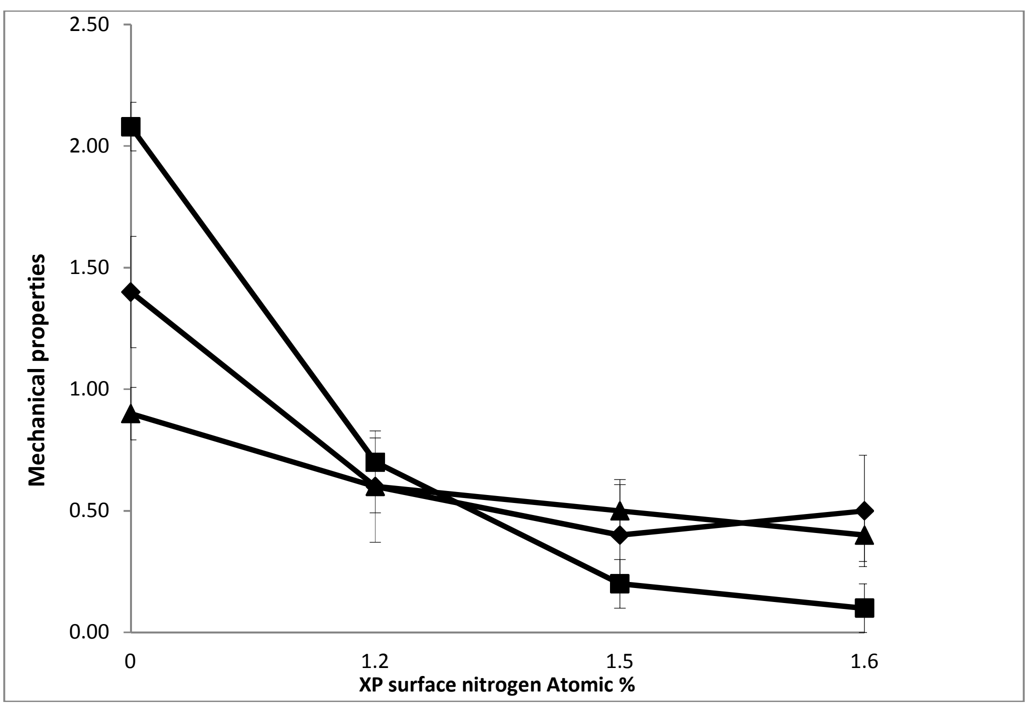

Fig. 6. Variation of mechanical properties with surface nitrogen assigned to DMDHEU of hand sheets made from DMDHEU treated cotton pulp
♦-*Tear strength-kN,* ▲-*Tensile index-kNm/g,* ■-*Tensile strength-kN/m*

The traditional paper strength is a function of ability of the fibres within the pulp to form hydrogen bonds to each other and the formation of these hydrogen bonds is via the cellulose hydroxyl groups, Fig. 7 and the higher the amount of the hydroxyl groups available for hydrogen bonding the stronger the paper.

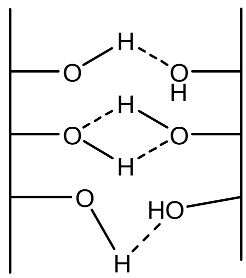

Fig. 7. Hydrogen bonding network among fibres within the hand sheet
| - polymer chains in the fibres

However following crosslinking within the cellulose polymer structure the easy care finished cotton fibres will have fewer free hydroxyl groups hence fewer or no hydrogen bonds formation among the fibres, Fig. 8. This explains the increase in Schopper-Riegler values with DMDHEU concentration were due the effects of shorter fibres and not surface properties of the fibres, subsection 3.1.

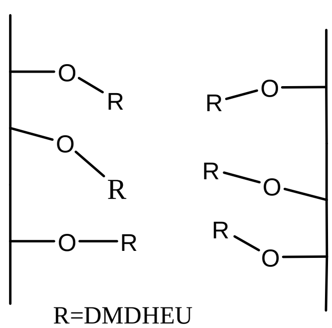

Fig. 8. The interaction between cellulose polymer and DMDHEU
| - cellulose polymer

Accordingly the hand sheet will be weakly formed as the concentration of DMDHEU increases in the pulp and may fail at relatively lower load. These two factors are supported by the decrease in the contribution of individual fibre to the strength of the hand sheet with the amount of crosslinked DMDHEU as determined by the tensile properties, Fig. 6.

3.3 The Effect of DMDHEU on KES-F Mechanical Properties of the Hand Sheets

The KES-F system was used to determine the effects of the easy care finishes on the properties of the hand sheets produced from reclaimed cotton pulp. Table 1 indicates the effects of incorporating DMDHEU into the cellulosic chains, as indicated by the surface nitrogen (determined by XPS) assigned to the chemically bound DMDHEU, on the bending (B) and shear rigidity (G) of the hand sheets.

The shear and bending rigidity of the sheets made from reclaimed cotton pulp decreased with increasing DMDHEU concentration in the hand sheet. The bending rigidity of the hand sheet is a measure of inter-fibre forces in the hand sheet, while the shear rigidity is a measure of inter-fibre frictional forces in the hand sheet. The decrease in the bending and shear rigidity of the hand sheets with increasing DMDHEU crosslinked to the cellulose polymer were due to the reduction of inter-fibre bending and shear forces caused by the DMDHEU crosslinking. The main forces holding the fibres together in the hand sheets are fibre-fibre hydrogen bonds but as explained, the forces are reduced due to reduced number of hydroxyl groups available for formation of the bonds Thus with the increase in the concentration of the DMDHEU the sheet can easily be deformed under shear or bending load. The effect of DMDHEU concentration on shear and bending properties of the hand sheet was more obvious at higher application levels, thus the sheet with surface atomic nitrogen of 1.6% could not withstand the bending and shear tests force and the results were not recorded, Table 1.

Table 1. Variation of bending and shear properties of hand sheets produced from DMDHEU treated pulp

Surface nitrogen (atomic %)	B (gfcm^2/cm)	G (gf/cm^o)
0	1.8±0.03	6.2±0.02
1.2	1.4±0.02	5.8±0.03
1.5	0.09±0.04	3.2±0.03
1.6	ND	ND

ND=Not detected

Compression properties such as compressional energy, (WC) and thickness measured at 0.5 gf/m^2, (T_o) and thickness measured at 50 gf/m^2, (T_m), of the hand sheets made from reclaimed cotton pulp treated with increasing DMDHEU

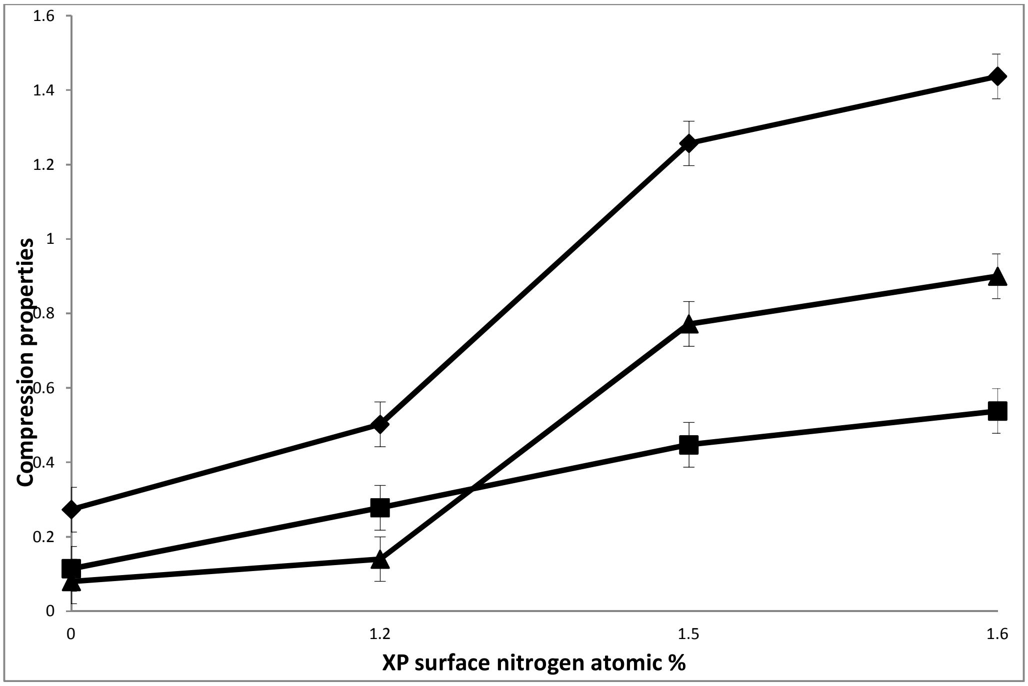

Fig. 9. Variation of compression properties with surface nitrogen assigned to DMDHEU of hand sheets made from cotton pulp treated with DMDHEU at increasing concentrations
♦-T_o-mm, ■-T_m-mm, ▲-WC-g.cm/cm^2

Table 2. Variation of surface properties of hand sheets produced from DMDHEU treated pulps.

DMDHEU Concentration(g/L)	MMD	SMD
0	0.0184±0.003	1.626±0.003
60	0.0106±0.002	1.352±0.002
100	ND	ND
140	ND	ND

ND= Not detected

concentration were also evaluated. The WC, T_o and T_m of the hand sheets increased with increasing DMDHEU crosslinked to the cellulose polymer, Fig. 9.

WC is the energy required to compress the hand sheet to a prefixed maximum load level and is related to sheet thickness (T_o and T_m) because the thicker the sheets the higher the energy required to compress it to the prefixed load. The increase in WC, T_o and T_m with amount of DMDHEU crosslinked with cellulose polymer was due to the shorter fibres produced under the effect of DMDHEU. The increased fines content and shorter fibres in the DMDHEU pulp produced fuller and thicker hand sheets than the DMDHEU-free hand sheet. The poor inter-fibre bonding in the hand sheets treated with DMDHEU also contributed towards the higher compression properties due to weak forces holding the fibres in the sheet.

The surface properties of the hand sheets as function of applied DMDHEU concentration were also affected by the presence of the DMDHEU easy care finish, Table 2 with both the mean deviation of coefficient of friction (MMD) and geometrical roughness (SMD) decreasing with increasing DMDHEU levels. This variation was explained by the ability of the fabric treated with DMDHEU to produce pulp with high fines and shorter fibres which form no curls and kinks in the sheet. Thus the surface roughness and harshness decreased with the increase in the DMDHEU concentration in the cellulosic fibres.

4. CONCLUSIONS

The effect of easy care finishes on the properties of pulp reclaimed from cotton garments was investigated. The cotton fabrics were treated with increasing concentration of DMDHEU and then deconstructed into pulp using Valley beater.

Fibre length and wetness properties of the pulp were determined. Furthermore, the pulp was made into hand sheets and the mechanical and hand properties of the sheets determined. The results have demonstrated that, the easy care finishes produced pulp with shorter fibres, high fines content but hand sheets with relatively weaker mechanical properties. Although the production of shorter fibres can be advantageous for the dissolution process, the bonded easy care finish appears to hinder the accessibility of the cellulose hydroxyl groups for hydrogen bond formation. This necessitate that in order to regenerate fibres from cotton based waste garments by the lyocell process, there must be an inclusion of the removal of the easy care finish as preparation stage for cotton waste garments. The stripping off the DMDHEU easy care finish may improve cellulose accessibility by solvents during the dissolution process.

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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