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Development of pretreatment solution for inkjet printing of polyester fabric using enzyme and cyclodextrin

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A pre-treatment solution has been developed for direct to fabric inkjet printing of polyester fabric with improved colour yield, colour fastness, and outline sharpness of the prints. Enzyme treatment of polyester fabric with lipase enzyme (*Candida Antarctica* B) has been carried out initially followed by treatment with cyclodextrin (20-60g/L) and citric acid (5-60g/L). The colour yield (*K*/*S*), and outline sharpness of inkjet prints on the pre-treated polyester fabric are evaluated. SEM, FTIR, and the water contact angle tests are conducted to study the utilization of the carboxyl group created by enzyme treatment for the attachment of cyclodextrin on fabric. The colour yield and sharpness of the pre-treated polyester fabric has improved as compared to the control and commercial samples used in the industry. The fabric samples pretreated with enzyme (1 g/L) followed by treatment with cyclodextrin (40 g/L) and citric acid (20 g/L) show higher colour strength and outline sharpness.

Keywords: Cyclodextrin, Inkjet printing, Lipase enzyme, Polyester

1 Introduction

With the growing demand for short-run lengths and a less expensive sampling process, the textile printing industry has adopted inkjet printing. The inkjet printing is done widely with four process colours CMYK (Cyan, Magenta, Yellow, BlacK). The correct firing of the fine, tiny droplets leads to a combined effect of the coloured image on the fabric with a wide range of colour gamut. Inkjet printing on textiles demands a suitable pretreatment on fabric for achieving better image quality in terms of colour strength and outline sharpness. The pretreatment acts as an intermediate layer between the textile and the inkjets (low viscosity) inks falling on them. It limits an ink drop from spreading, controls it from wicking through the macro capillaries between the yarns and prevents the uncontrolled diffusion of the ink inside the fibres. The type of pretreatment depends on fibre and accordingly on the substantivity of the ink towards the fibre. The pretreatment of cotton fabric is made with sodium alginate traditionally and this is available commercially. Alginate is found to be the best pretreatment solution for cotton fabrics and they are available commercially with other ingredients like urea, and sodium bicarbonate. The alginates can also be used for the pretreatment of polyester fabric[1], but the adhesion is limited due to the hydrophobic nature of the fibre. To improve the hydrophilicity, studies have been carried out using plasma and enzyme as surface modification techniques[2-10]. Plasma treatment is one of the most widely used techniques for the introduction of polar groups on the surface, thereby imparting hydrophilicity to the fibre. Different plasma treatments by varying plasma generators and their gas like pure air, argon, oxygen and their mixtures, etc are studied on polyester fabric[2,3,5]

Similar to plasma, enzymes[4,6-10] are also used for surface modifications, such as bio-scouring, de-sizing and as an ingredient in detergents by the textiles industry. For inkjet printing pretreatment, enzymes are used for enhancing the treatment effect with sodium alginate. Ibrahim and Salam[6] have reported the use of enzymes as a pretreatment agent in digital printing. An increase in colour strength of about 10-30% due to enzyme treatment has been reported. Hennawi and Shahin[7] treated linen, polyester fabrics and their blends with enzymes from cellulose and yeast extract to enhance the effect of inkjet printing on the fabric.

Further studies on naturally occurring agents, like chitosan, cyclodextrin, xanthan gum and their derivatives are reported[11-17]. The study on the modification of chitosan and its ability to improve the colour yield of cotton fabric was carried out by chen and

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wang[13] with pigment inks. Grafting of glycidyl trimethyl ammonium chloride to amino groups of chitosan resulted in the formation of 3-Chloro-2- hydroxy propyl trimethyl ammonium chitosan chloride (HTACC). The water soluble HTACC increased the colour strength (*K*/*S*) from 3.20 (untreated) to 4.87(HTACC pretreated) samples. Nappakundilograt and Bulanagul[11] has pretreated polyester with another modified chitosan named DBIC (dimethyl (aminobenzyl) imino) chitosan and HTACC. The water-soluble chitosan HTACC has given excellent properties over the rest of the pretreatment and only 0.1% (w/v) of HTACC was required to provide a high colour gamut, colour saturation and outline sharpness.

Cyclodextrin (CD)[17-20] is a polysaccharide that is used in the textile domain because of its complex forming properties. Once the CDs are grafted onto the textile they present special functionalities to the fibre or fabric. The CD grafting reaction by polycarboxylic acid is done which is similar to the reaction between cotton and BTCA. CD-finished textiles capture organic molecules, like fragrance, aromatic pollutants or drugs, through their inclusion into the grafted CDs cavities[18]. The grafting of cyclodextrin onto polyester fabrics is carried out by Ghoul and Mastel[19] using the intermediate of polycarboxylic acids as cross-linking agent and sodium dihydrogen hypophosphite as a catalyst. The polymer which is formed as a result of the reaction between the CD and a carboxylic acid compound will physically adhere or will entangle into the fibrous network.

Chen and Wang[13] has improved the sharpness and the colour yield of the polyester fabric with β-cyclodextrin as pretreatment. PET fabric is grafted with β-cyclodextrin (100g/L) and citric acid (100g/L). The pretreatment has enhanced colour yield (*K*/*S*) by 47% compared to the untreated samples. The line width also decreased by 77% and 62%. This improvement in image quality depends on the amount of polymer grafted on the fibre. The present study aims at investigating the effect of two bath pretreatment process using enzyme and cyclodextrin. Further, the image quality of inkjet printing in terms of area of spreading and outline sharpness is also assessed and discussed.

2 Materials and Methods

2.1 Materials

Commercially available 100% polyester fabric with plain weave (70gsm) was used for all the pre-treatment processes. β-cyclodextrin (CD) was supplied by Jay Chemical Marketing, Mumbai. The citric acid (CA), potassium dihydrogen phosphate (KH_2PO_4), dipotassium hydrogen phosphate (K_2HPO_4), sodium chloride, potassium iodate, potassium iodide and labolene were purchased from SRL Chemicals, Mumbai. Sodium hypophosphite (SHP) was purchased from Sigma Aldrich. Lipase enzyme (Candida antarctica B) was purchased from Fermenta Biotech Ltd., Mumbai. Inkjet inks (cyan, magenta, yellow and black) manufactured by KIIAN Digital, was provided by Metro-imaging, Chennai.

2.2 Methods

2.2.1 Enzyme Treatment of Polyester Fabric

The polyester fabric was treated with lipase enzyme *Candida antarctica* B (CALB) with varying concentration (1, 1.25, 1.5, 1.75, and 2 g/L). The material-to-liquor ratio was fixed at 1:30. The optimum reaction conditions for CALB are *p*H 7.5, and temperature 40□C as provided by the supplier and the reaction time was fixed at 30 min. The *p*H of the bath was maintained using 50 mM phosphate buffer. The treatment was carried out using a shaking water bath at 110 rpm and the enzymes were denatured by raising the temperature of the bath to 80□C for 10-15 min. The samples were then subjected to hot wash (60□C) followed by cold wash (room temp.) and then dried at 30□C

2.2.2 Pretreatment using Cyclodextrin

The enzyme-treated fabrics were treated with various concentrations of CD and CA, keeping material to liquor ratio fixed at 1:30. The CD and CA were taken in the ratios of 1:1, 1:0.5, and 1:0.25. The concentration of SHP was kept constant at 30g/L. Weighed quantities of CD and SHP were taken in a beaker containing water and placed in a magnetic stirrer until the solution attains 80 □C. Upon complete dissolution of CD in the solution, CA was added. The prepared CDCA solution was padded on to the enzyme-treated fabric and the wet pickup was adjusted to 70%. The fabric was then dried at 80 □C for 10 min and cured at 160 □C for 6 min. The codes given for the samples treated with different concentrations of CD and CA are given in Table 1.

2.2.3 Drop Test on Pre-treated Samples

As a preliminary test for finding ink drop spreading on untreated and pretreated fabrics, the drop test was carried out with the help of a micro-pipette. The

Table 1 — Different concentrations of CD and CA and their codes

CD:CA :: 1:1	**ECD20CA20**	**ECD40CA40**	**ECD60CA60**
Enzyme, % owf	1	1	1
CD concentration, g/L	20	40	60
CA concentration, g/L	20	40	60
CD:CA :: 1:0.5	**ECD20CA10**	**ECD40CA20**	**ECD60CA30**
Enzyme, % owf	1	1	1
CD concentration, g/L	20	40	60
CA concentration, g/L	10	20	30
CD:CA :: 1:0.25	**ECD20CA5**	**ECD40CA10**	**ECD60CA15**
Enzyme, % owf	1	1	1
CD concentration, g/L	20	40	60
CA concentration, g/L	5	10	15

fabrics were printed with drops of 100% ink. The volume of 2μL was found to produce minimum error in the micropipette, and the measurement line in the tip coincided with the volume of 2 μL chosen. This helped in the identification and correction of the malfunction of the pipette and manual errors during printing. The printing was carried out using all the inks, namely cyan, magenta, yellow, and black (CMYK). The printed samples were dried at 80 °C for 5 min and cured at 210 °C for 1 min as per the ink manufacturer's recommendation. After printing, all samples were subjected to reduction clearing using soap at 60°C. It was followed by immersion of the printed fabrics in sodium hydroxide (2g/L), sodium dithionite (2g/L) for 15-20 min at 65 °C. The samples were then hot-washed (60 °C) followed by cold-wash (30 °C) and dried at room temperature(30 °C). Assessment of the effect of pre-treatment on print quality was carried out. The print quality was measured in terms of area of spreading. The drop tested samples were scanned using HP scanner and then the scanned images were analysed using ImageJ software. The measurements were taken in pixels and it was confirmed using a scale tool in the software that a dot in an image was equal to a single pixel value of the scanned image. The images were then inverted into 8 bit binary image. The values were taken for three replicas of the sample and the mean value was calculated.

2.2.4 Printing of Fabric using Commercial Inkjet Printer

Inkjet printing was carried out on commercial inkjet digital printer by Mimaki with Epson Dx5 print head at resolution of 600 dpi. The printed samples were dried at 80 °C for 5 min and cured at 210 °C for 1 min. The samples were printed with two different line widths of 1 point and 8 point on both warp and weft directions (Fig. 1).

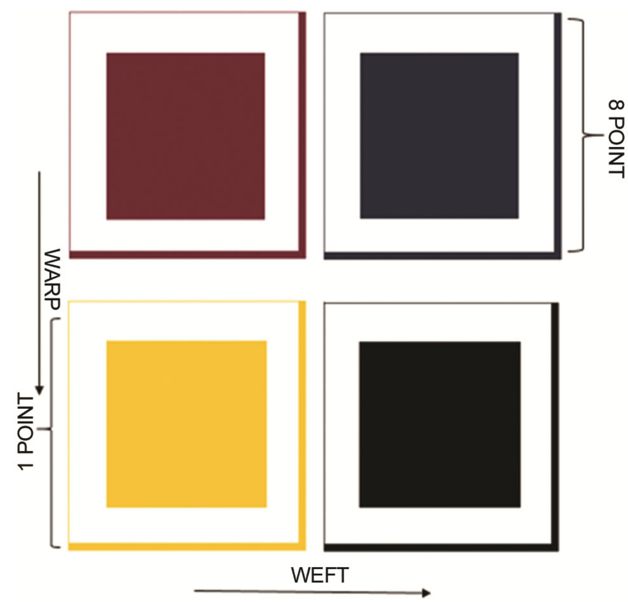

Fig. 1 — Print design with CMYK colours having two print widths

2.2.5 Measurement of Outline Sharpness

The printed widths were collected from optical microscope (Labomed, LX-300) images. The printed images were analysed using ImageJ software. The images were then inverted into 8 bit binary image. The printed widths in both warp and weft directions were calculated. The values were taken for three replicas of the sample and the mean value was calculated.

2.2.6 Measurement of Colour Yield

The colour strength of the samples was measured using Techkon Spectrophotometer. This machine has an aperture size of 3 mm and it determines reflectance values for every 20 nm wavelength from 400 nm to 700 nm. The measurements were taken with 10° absorber and D65 lamp source. The measurements were taken in two separate regions of the sample and the readings were averaged. One reading was taken at the centre and another at the edge of the printed samples. The reflectance curve was plotted for every sample and for all the four colours (CMYK) used for the study. The *K*/*S*, L*, a*, b* were calculated for all the samples using Kulbeka-Munk's equation.

2.2.7 Colour Fastness of Fabric

The colour fastness values of the printed samples were analysed for wash, light and rubbing tests. The wash fastness was tested using AATCC test method 61-2001 in Laundrometer (R.B.Electronics & Engineering Pvt. Ltd). The rub fastness was tested using AATCC test method 8-2001 in Crockmeter (MAG Solvics Pvt Ltd.,). Wet and dry rubbed samples were compared against ISO standard for staining and color change. The light fastness test was carried out using ISO105 B02 in Digi-Light (Paramount Instruments Pvt. Ltd).

2.2.8 SEM and FTIR Studies

In order to study the surface roughness and morphology before and after pretreatment, the surface morphology of the fabrics was analysed using micrographs obtained from TESCAN VEGA 3 SEM. The difference in surface morphology of the untreated fabric (control), enzyme-treated fabric and cyclodextrin-treated fabric was analysed. Fourier transform infrared spectroscopy data was obtained from Thermo Nicolet 6700 FTIR Spectrometer (Thermo Fisher Scientific) for the study.

2.2.9 Measurement of Water Contact Angle

Water contact angle (WCA) measurements were taken using Goniometer from Jinan Cyeeyo Co, Ltd. The increase in hydrophilicity after enzyme treatment was compared against untreated polyester fabric. Further, the changes in WCA of cyclodextrin-treated samples was also studied.

3 Results and Discussion

Polyester fabric is known for its hydrophobicity. Introducing hydroxyl and carboxyl groups in polyester fabric through lipase enzyme treatment has been reported by number of researchers[7-10]. Due to enzyme treatment, a decrease in water contact angle and increase in hydrophilicity of the PET samples are observed by Lee and Song[9].

3.1 Effect of Enzymatic Hydrolysis of PET Fabric on Image Quality

Trials have been conducted to study the effect of enzyme hydrolysis of PET with varying enzyme concentrations (1, 1.25, 1.5, 1.75, 2gpl). The drop test reveals that control PET fabric (untreated) have higher spreading in the warp direction than in the weft direction (Fig. 2). This results in lower colour intensity of untreated fabric as compared to the enzyme-treated fabric. The amount of ink penetration in the warp direction is found higher as compared to that in the weft direction in control fabric. With enzymatic hydrolysis, the difference in ink penetration in the warp and weft directions is minimised (Fig. 2)

This is due to increase in hydrophilicity of the fabric. This is confirmed by the water contact angle measurements taken on untreated and enzyme-treated polyester fabrics. There is a decrease found in water contact angle of the enzyme treated fabric (99□) from untreated PET (118□) as shown in Fig. 3. This is in agreement with the study where CALB[9] decreases the WCA of the PET from 95.7^0 to 67.7^0. Further, the decrease in print area may be due to the increase in hydrophilicity of the fabric (Fig. 4)

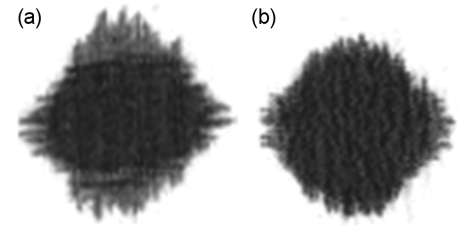

Fig. 2 — Drop spreading in (a) control polyester fabric and (b) enzyme treated polyester fabric

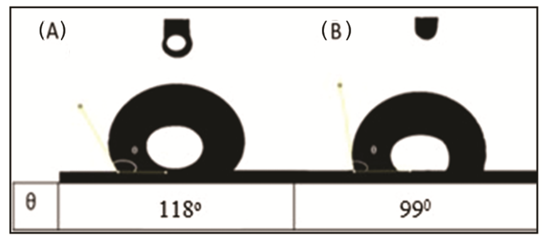

Fig. 3 — Water contact angle of (A) control fabric and (B) enzyme-treated fabric

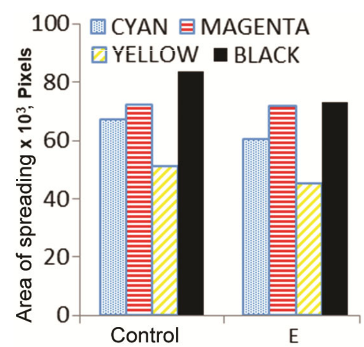

Fig. 4 — Comparison of image qualities of control fabric and enzyme-treated (1g/L) fabrics (E)

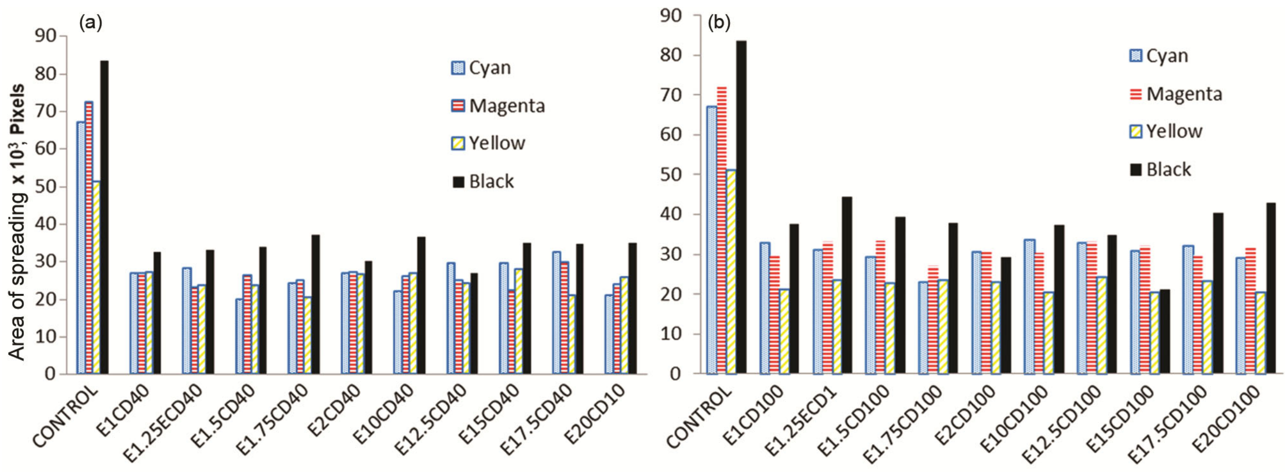

Fig. 5 — Area of spreading measured for different concentrations of enzyme with cyclodextrin (A) 40g/L and (B) 100g/L

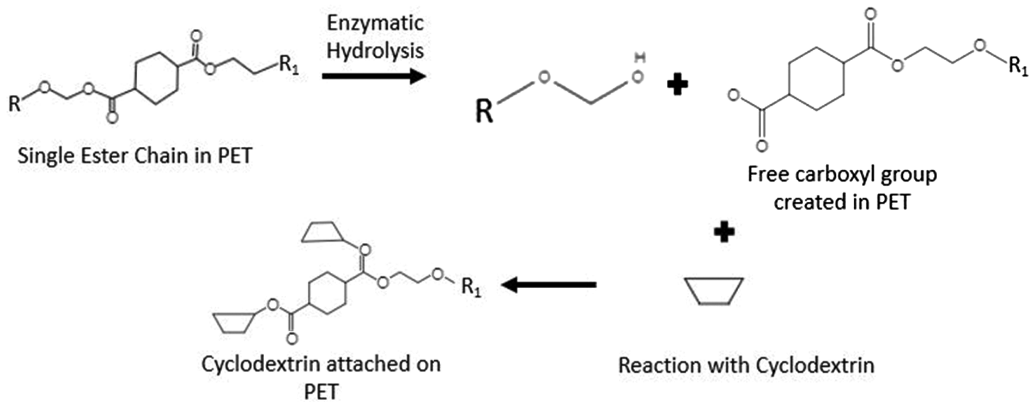

Fig. 6 — Proposed reaction mechanism for attachment of CD on enzymatically hydrolysed polyester fabric

3.2 Role of Enzyme in Fixation of CD

In this study, trials have been conducted for studying the effect of enzyme hydrolysis of PET with varying lower enzyme concentrations (1, 1.25, 1.5, 1.75 and 2g/L) and higher enzyme concentration (10, 12.5, 15, 17.5 and 20g/L). Two concentrations of cyclodextrin and citric acid in (1:1) ratio with 40g/L and 100g/L are taken. The fabric treated with enzyme followed by CD has lower area of spreading as compared to untreated and enzyme-treated fabrics. No significant difference is observed between the samples for cyan and magenta colours in terms of (PA) as shown in Fig. 5. To obtain uniform spreading of all the colours, the samples with minimum difference in area of spreading for black and yellow colours are found to be E1CD40, E2CD40, E12.5CD40 on lower range and E2CD100, E15CD100 on higher range. Among different concentrations of enzymes used for the study, 1g/L of enzyme concentration gives higher image quality. Hence, a lower concentration of enzyme (1g/L) is selected for further studies. Further, as it can be seen in Fig. 5, lower concentration of CD (40g/L) controls spreading as compared to higher concentration (100g/L). It is expected that the increase in image quality is due to the reaction between carboxyl groups created on the PET fabric by enzyme treatment with CD. The proposed reaction mechanism is given in Fig. 6.

3.3 Utilization of Carboxyl Groups created through Enzyme Treatment for Minimization of PCA in Grafting of CD

Permanent grafting of CD on PET with polycarboxylic acids (PCA) reveals that polyesterified products are formed with the carboxylic acids like citric acid (CA) cured at 160^{0}C. An optimum ratio of 1:1 of CD:CA is required for forming polyestersified product. Previous work carried out by Chen[13] has improved the sharpness and the colour yield of the polyester fabric with cyclodextrin (100g/L) and citric acid (100g/L). Citric acid has disadvantages like degradation of fibre and might interact in original color of the ink. Hence, to reduce the effect of CA, the concentrations of CA is reduced and enzyme treatment is carried out prior to CD

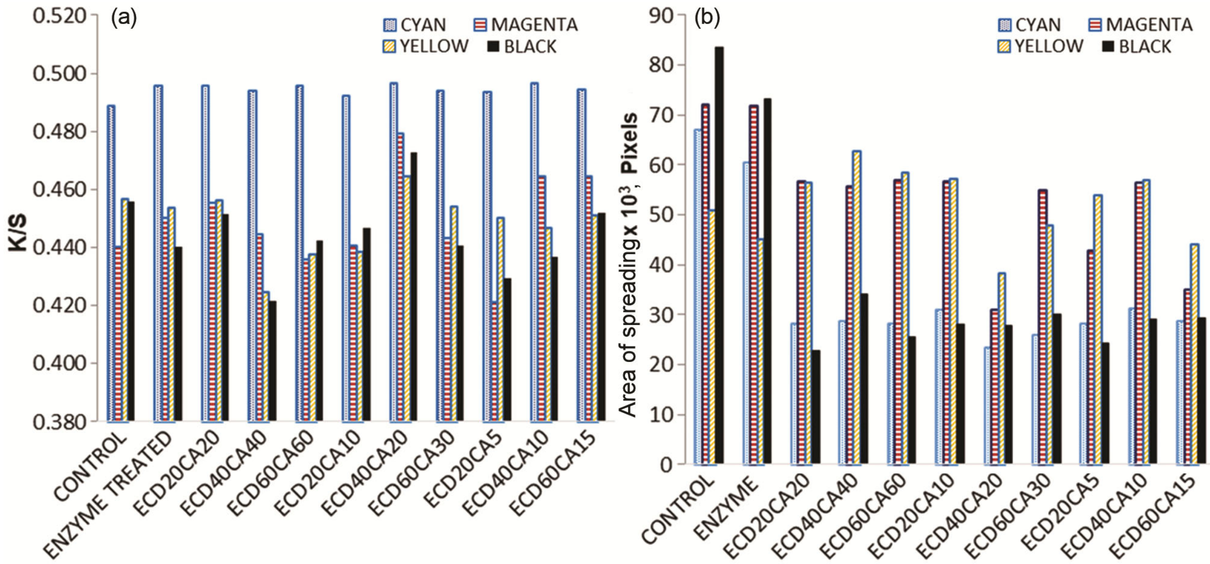

Fig. 7 — Comparison of drop tested samples for (a) colour strength (*K*/*S*) and (b) area of spreading

attachment. The CD and CA are taken in the ratio of 1:1, 1:0.5, and 1:0.25. The concentration of SHP is kept constant at 30g/L.

3.3.1 Measurement of Color Yield

Drop test is carried out for all the samples. Figure 7(a) shows the colour strength (*K*/*S* value). On comparing all the samples for cyan colour, no significant difference is noticed. The highest colour strength is obtained for ECD40CA20 for magenta, yellow and black colour, except for cyan. The sample ECD20CA5 has poor colour strength for all the colours. This indicates that very low concentrations of CA might result in poor adhesion of CD on polyester fabric. Hence, the optimum concentration of CA for attachment of CD is considered as 10g/L.

3.3.2 Measurement of Area of Spreading

The area of spreading of the drop tested samples is measured using ImageJ software and the results are given in Fig. 7 (b). As it can be observed from the graph, the area of spreading varied from colour to colour. On comparing all the colours, the area of spreading is lowest for the sample ECD40CA20 for the cyan, magenta and yellow colour. However, the area of spreading for black is lowest for ECD20CA5 and ECD20CA20, but a comparable reduction in spreading is observed for EC40CA20 sample. Hence, the sample ECD20CA20 provides a uniform lowest area of spreading for all the colours.

3.4 Comparison of Image Qualities

The best sample ECD40CA20 is taken for inkjet printing in commercial inkjet printer and the results are compared against two commercially available pretreated polyester fabric with 70 and 110 gsm respectively.

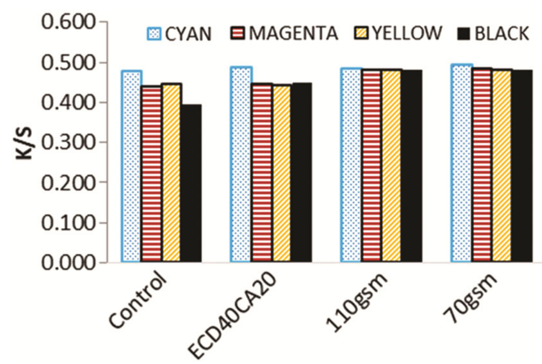

Fig. 8 — Colour strength (CMYK colours) of the printed samples

3.4.1 Measurement of Colour Strength

Colour strength has been measured for the sample ECD40CA20 solution and commercial samples obtained from the industry. It is revealed that except for cyan, ECD40CA20 has a higher colour strength for all other colours (magenta, yellow and black). Black colour shows a higher colour yield followed by yellow, magenta and cyan (Fig. 8).

3.4.2 Outline Sharpness

Outline sharpness was measured for ECD40CA20 and the two commercial samples. The printed images with thick and thin lines on ECD40CA20 is shown in Fig. 9 (a) and the area of spreading in terms of pixels is shown in Fig. 9 (b). It is evident that ECD40CA20 shows a better and equivalent sharpness like commercial sample

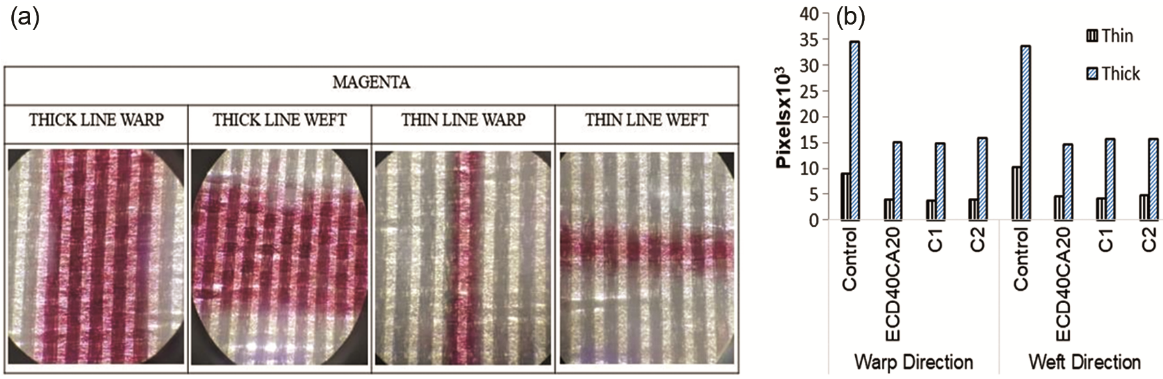

Fig. 9 — ECD40CA20 sample (a) images of prints with thick and thin lines and (b) outline sharpness in warp and weft directions

Table 2 — Colour fastness results of printed samples

Sample	Light C/M/Y/K	Crocking (C/M/Y/K)		Washing						
				Staining (C/M/Y/K)						Color change (C/M/Y/K)
		Dry	Wet	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
Control	7/7/7/7	4/4/4/4	4/3/4/3	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4/4/4/4	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4/4/4/4
ECD40CA20	7/7/7/7	4/4/4-5/4	3/3/4/3	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4-5/4-5/4/4-5	4-5/4-5/4-5/4-5
110 GSM	7/7/7/7	4/4-5/4-5/4	3/3/4/3	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4-5/4
70 GSM	7/7/7/7	4/4-5/4-5/4	3/3/3/3	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4/4	4/4/4-5/4

3.4.3 Measurement of Colour Fastness

Measurement of colour fastness was carried out for different samples with various concentrations of cyclodextrin (20-60g/L) and citric acid (5-60g/L). The colour fastness rating of inkjet-printed polyester fabric is given in Table 2. As it can be seen from the results, the colour fastness to rubbing, light, and laundry, the pre-treated fabric (ECD40CA20) is comparable with the commercially available fabric.

3.5 Study of SEM

The scanning electron microscopy images are taken for control, enzyme treated and the sample ECD40CA20. Fig.10 shows the SEM micrograph of untreated polyester fabric to have a smoother surface without roughness. The presence of small holes and crevices in the surface of enzyme-treated fabric can be seen in micrograph. These small holes and crevices can help in peneretation of cyclodextrin and ink after enzyme treatment. The lipase enzyme treatment can significantly improve the water penetration and absorbent property of polyester fabric[7] Further, the sample ECD40CA20 shows the cyclodextin forming patches on the PET fibre and implies that the pretreatment solution has adhered to the fabric surface and creates a rougher surface on PET fabric.

3.6 Study of FTIR

FTIR spectra is taken for controlled fabric, enzyme-treated fabric and ECD40CA20 sample. The results are shown in Fig. 11. PET sample shows the characteristic peak at 1721cm^{-1}, which is the peak absorption of polyester. Enzyme treated sample shows characteristic peaks at 3550-3450 cm^{-1} and 1736 cm^{-1}, which is assigned to the O-H stretch &–CO group and ester group respectively. Sample ECD40CA20 shows the characteristic peak at 1627 cm^{-1}, which assigns the cyclodextrin.

3.7 Determination of Tensile Strength

Measurement of tensile strength is carried out for control fabric, enzyme-treated fabric, (ECD40CA20 sample) in terms of B-Force (kgf). Tensile strength in warp direction is found higher than in the weft direction for all the samples. Upon treatment with enzyme, there is a decrease in the strength of the polyester fabric from 53.7 kgf to 50.86 kgf in warp direction and from 35.10 kgf to 33.06 kgf in weft direction. Further treatment with cyclodextrin has resulted in further decrease in tensile strength in weft direction to 30.02 kgf. But in the warp direction an increase in tensile strength to 54.73 kgf is observed. The decrease in tensile strength may be due to

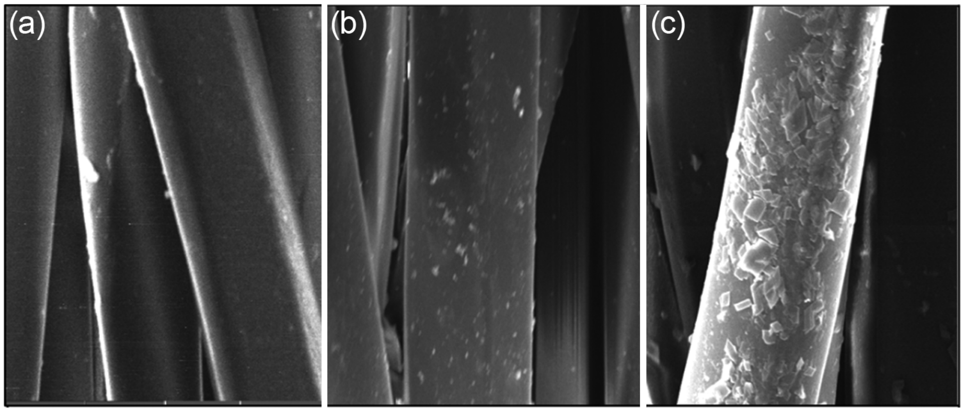

Fig.10 — SEM images of (a) control fabric, (b) enzyme treated fabric and (c) ECD40CA20

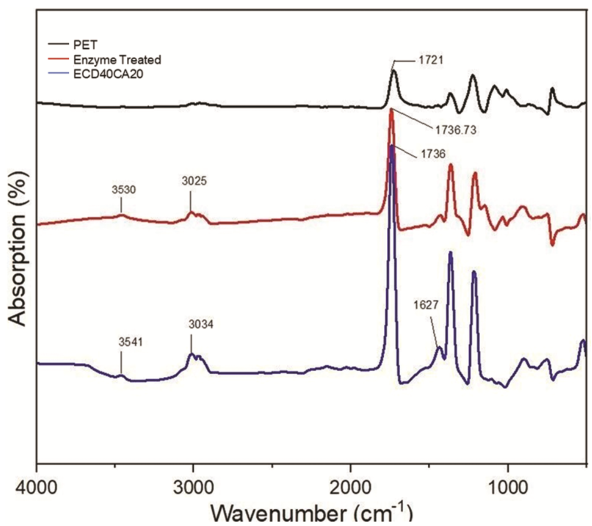

Fig. 11 — FTIR spectra of control fabric, enzyme-treated fabric and ECD40CA20

degradation of fabric owing to enzyme and citric acid used in the pretreatment solution.

4 Conclusion

Pre-treatment with β- cyclodextrin (CD) shows better results for colour strength and colour fastness. Citric acid (CA) helps in forming carboxyl groups and acts as a crosslinking agent, which helps in forming a bond between polyester and CD. But using CA at higher amount results in the yellowing of the fabric. So, enzyme treatment is carried out before pre-treatment with CD as it also helps in forming carboxyl (–CO) groups. Further, these carboxyl groups can be utilized for CD to adhere on polyester fabric, thereby reducing the amount of citric acid in the pre-treatment process. The fabric pre-treated with enzyme followed by treatment with CD at 40 g/L and CA at 20 g/L (ECD40CA20) gives higher image quality both in terms of area of spreading and colour yield as compared to other samples. The SEM and FTIR results also confirm the formation of ester linkages on treated polyester fabric (ECD40CA20). Further, on comparing with commercial samples from industry, the treated polyester fabric (ECD40CA20) has a comparable image quality. Tensile strength results reveal that the strength of the pretreated samples is reduced compared to untreated fabric. Further works are in progress to reduce the effect of pretreatment on tensile strength of fabric.

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