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Khawar, MT orcid.org/0000-0001-7150-0465, Tausif, M orcid.org/0000-0003-0179-9013, Ashraf, M et al. (4 more authors) (2021) An experimental study on dyeing of needle-punched polyethylene-terephthalate non-wovens. *Coloration Technology*. ISSN 1472-3581

<https://doi.org/10.1111/cote.12533>

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An experimental study on dyeing of needle-punched polyethylene-terephthalate nonwovens

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Abstract

The dyeing of nonwovens in fabric form is of interest for a range of applications especially aesthetics. In this work, needle-punched nonwoven fabrics were prepared by employing polyethylene terephthalate (PET) fibres of two different linear density and same mean fibre length in four different fiber linear density ratios and three different fabric mass areal densities, while keeping all other process and material variables constant. The objectives of the study were to find out the saturation point in the dyeing profile of needle-punched nonwovens by varying aforementioned process variables and, for the first time, compare different industrially relevant conventional fabric dyeing methods (Jet, Package and Jigger dyeing) in terms of physical, mechanical and colour fastness properties. In case of Jet and Package dyeing, nonwoven fabric was successfully dyed without any structural reorientation while fabric sample deformation is reported in jigger dyeing process. Furthermore, tensile strength (N) of samples was increased, while air permeability (mm/sec) was decreased due to the slight compactness of structure after dyeing process.

Key words: dyeing, nonwovens, needle-punched, industrial, polyethylene terephthalate

1 Introduction

Nonwovens are directly manufactured from fibres/filaments whereas man-made fibres (such as polypropylene, polyester (PET) and viscose) are pre-dominantly employed for the production of nonwoven fabrics. The nonwoven manufacturing includes web formation (dry-laid, wet-laid, and polymer-laid) and web bonding (mechanical, thermal or chemical means) methods ¹. Finishing by using both dry and wet methods is integral part of many nonwovens fabrics for functional as well as aesthetic purposes. The finishing of nonwovens is totally dependent on the required features in the end product. Majority of the finishing processes such as, dyeing, washing, calendaring and printing have evolved from the conventional textile processing industry ².

Nonwovens are employed largely in all the sectors of the technical textiles. At their own or in combination with other conventional materials, these are being used in a wide range of products for a myriad of applications; both durable and limited life products ³. The colouration of nonwovens find application in various fields including, floor coverings, wallpapers, furnishings, table and bed linen, shoe linings, as well as single- or limited-use protective clothing interlinings ⁴. Garment interlinings are also often coloured. In general, there are several methods for the dyeing of all fabrics; the continuous method, mass pigmentation method, Pad Batch method (semi continuous) and the batch dyeing method. Another approach is the use of dope dyed fibres for the production of coloured nonwovens ². Nonwovens are inherently different from knitted and woven fabrics owing to direct conversion of fibres/filaments to fabrics.

Mass-pigmented polypropylene is widely being used for the nonwovens manufacturing ⁴. In this technique, colourant master-batch mixed with the polymer in melting phase, prior to

extrusion which provides better consistency as compared to other techniques ⁵. Pigment application is a versatile method for colouration of nonwovens with the help of binder application. Uniformity of the applied suitable binder is very important for the uniformity of colouration because application of pigmented binder can reorganize on the substrate during drying due to differential migration of solids to the solvent evaporation boundary ⁶ and can significantly affect the handle of the fabric. By using pigment printing technique, PET nonwoven showed excellent shade depth and significant enhanced tensile properties. This process can be used to improve the aesthetic factor and can also be applied as a finishing treatment of the bonded nonwoven webs ⁷.

Nonwoven fabric can also be dyed by using conventional methods either by continuous method or batch dyeing method. Continuous process of dyeing involves high tension during dyeing because fabric movement is totally dependent on the drives of guide roller for fabric movement from one bath to other openly. The dimensional stability and modulus of nonwoven fabrics varies depending upon the raw materials and method of manufacturing. Hence, the application of colour through continuous method is dependent on the fabric structure. Batch dyeing method is more efficient to achieve the uniform shade of the dye which enhances the quality of the dyed fabric. In batch dyeing, the movement of fabric and/or liquor results in different dyeing technologies ². PET fiber is also widely produced in textile sector and is also being used to make nonwoven fabric. PET fiber has low moisture regain and non-polar group on the surface. Dyeing of PET fiber is typically performed at high temperature (130°C) and high pressure, due to its low hydrophilicity and high crystalline structure ⁸.

Burkinshaw et al. ⁹ compared the dyeing of cotton and polyethylene terephthalate (PET) hydro entangled nonwovens to that of woven and knitted PET fabrics by using pot dyeing machine. The porous structure and large void volume in hydro entangled nonwovens as

compared to woven and knitted fabrics, is known to aid access of dye molecule to fibre surface and gives higher K/S output. The results showed that K/S of nonwovens was comparatively greater than that of knitted and woven fabrics (nonwovens > knitted > woven). It was observed that dye uptake was dependent on the construction of fabric rather than the dwell time, fibre type or specific class of dyes. It was also reported that higher porosity of nonwovens allowed the dye molecules to penetrate easily, as compared to knitted and woven fabrics.

The effect of conventional technique of dyeing and dope dyeing of polyamide nonwoven web has been investigated by the Mayer et al. ¹⁰. This work illustrated the importance for the applications as pH sensitive sensor materials. Dope dyeing at acidic pH shows the leaching of dye. Canbolat et al. ¹¹ dyed the PET/viscose blended fabric using oxygen plasma treatment and found that treated fabric has improved color strength (K/S) as compared to untreated fabrics.

There is a paucity of published scientific literature on dyeing of nonwovens and to authors' knowledge, there is no published work on dyeing of needle-punched nonwovens. The current study aims to analyse the effect of fibre linear density and fabric areal density on K/S of dyed nonwovens, and also, for the first time, compare various industrially relevant dyeing methods to determine suitable dyeing method of needle-punched nonwovens on the basis of physical, mechanical and colour fastness properties.

2 Materials and Methods

2.1 Nonwoven Fabrics Samples

PET (Polyethylene Terephthalate) fibres of two different linear densities and same mean fibre length were used in four fiber linear density ratios and fabric areal densities to produce 12 samples. Fibre properties of chosen PET are given below in Table 1.

Table 1: PET Fibre Properties

Description	Fibre Length, mm	Fibre Strength cN Tex ⁻¹ (gpd)	Elongation, %	Crimp cm ⁻¹
PET 1.5 denier	76	37.43 (4.23)	67.0	3.87
PET 3 denier	76	37.96 (4.29)	69.9	3.39

The needle-punched fabrics were prepared by keeping all other process and material variables constant. The PET nonwoven fabric was prepared on needle-punching machine (Dong Wong Roll DW-N/P) which was equipped with fine opener, reverse hopper feeder, mini carding machine, cross lapper and needle punching machine. The PET nonwoven fabric was prepared in two steps which involved the formation of web on mini card machine, then the bonding of the web was performed on needle-punched machine. The needle punching was done at 300 rpm with delivery speed of 0.9 m/min. After needle-punching, the calendaring of nonwoven fabric was also done to compact the fabric structure. The nonwoven fabric's areal density was adjusted at 200 g/m². All twelve samples were initially opened well to prepare an even feed to roller-stripper carding machine followed by cross-lapping and needle-punching. The sample details are given in Table 2.

Table 2: Prepared nonwoven samples details

Sample ID	Mass areal density (g m ⁻²)	Fiber Linear Density ratio (1.5/3 denier)
1	100	75/25
2		50/50
3		25/75
4		0/100
5	150	75/25
6		50/50

7		25/75
8		0/100
9	200	75/25
10		50/50
11		25/75
12		0/100

All of the samples were heated at 185°C for one minute on lab scale stenter to avoid shrinkage during subsequent processes. The samples were scoured at 85°C for 15 min using 1 gL⁻¹ scouring agent (Rucogen WBL®) to remove lubricants and surface impurities from the samples.

2.2 Preparation of nonwoven fabric for dyeing

The preliminary work on dyeing profile and % owf (on weight of fabric), saturation point was planned to inform the conditions for subsequent dyeing of all the fabrics in Table 2. PET fibers are normally dyed by using disperse dyes and process of dyeing is called disperse dyeing¹². In addition to disperse dyes, whole process is performed with different chemical auxiliaries such as dispersing agents, penetrating agents and levelling agents in the dyebath under aqueous conditions^{13,14}. As per dyeing profile (Figure 1), Sample 10 (Table 2) was dyed (employing dyestuff Foron Black RD-SL) according to a temperature range of variables (50, 65, 80, 95, 110 and 130 °C) with time variables (10, 20, 30, 40, 60, 80 minutes) and K/S was measured to find the saturation point. To achieve the saturation point % owf, sample 10 (Table 2) was dyed for a range of 0.1% to 10% and measured for K/S. The dyeing experiments were performed using a pot dyeing machine (Datacolor AHIBA IR.), unless mentioned otherwise. The dyeing of all the samples was performed (Figure 1) with liquor

ratio of 25:1 in the presence of 1 g/L Dispergator XHT (ARCHROMA) as a dispersing and levelling agent. The samples then removed form dyeing bath and subsequently, all the samples were reduction cleared at 95 °C for 20 min using 2 g/L caustic soda (50% analytical grade) and 2 g/L sodium hydrosulphite (BASF). At the end, samples were neutralized with the help of citric acid (Monohydrate) 1.5 g/L at 50 °C temperature to maintain neutral pH. Sirrix 2UD (ARCHROMA) was used as a sequestering agent for the reduction of tap water TDS (Total dissolved solids) up to 300 ppm ¹⁵.

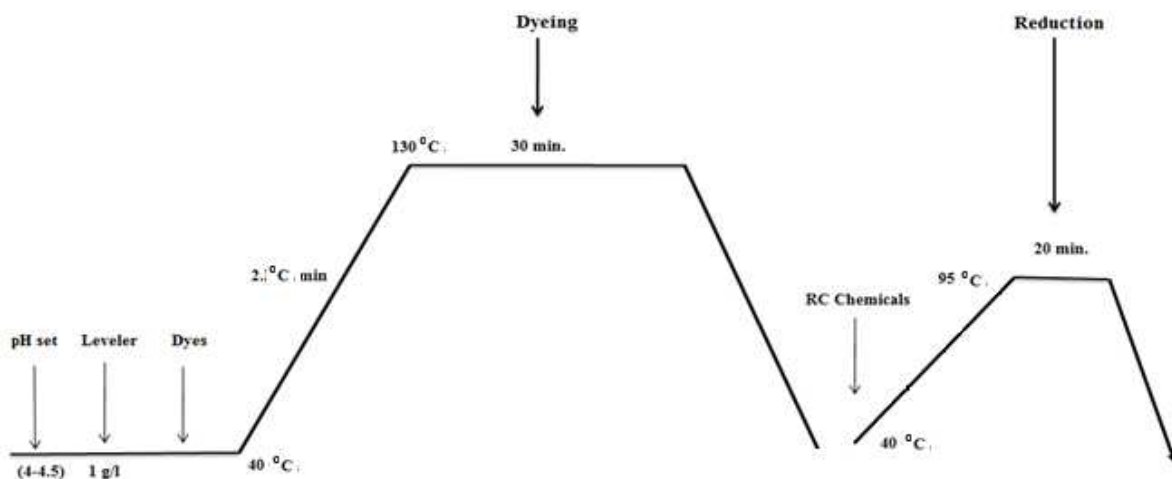


Figure. 1 Dyeing profile for the experimental work

Based on the conditions informed by the preliminary work, all subsequent dyeing of the fabrics in Table 2 was studied in three parallel stream of work.

- Effect of linear density of PET fibers on K/S (Samples 9, 10, 11, 12 – Table 2). All samples were 200 g m⁻².
- Effect of mass areal density on K/S (Sample 4, 8, 12 – Table 2). All samples were made from 100% 3 denier fibres.
- Effect of industrially relevant dyeing methods, on the basis of movement of fabric and/or liquor, on the K/S and fabric performance - Table 3.

Table 3: Experimental design for the selection of best dyeing method

Input variable		Output variables				
Machine type	Methodology (Movement)	(K/S)	Tensile Strength (N)	Air permeability (mm/sec)	Colourfastness to washing	Colour fastness to Rubbing
Jigger (Werner Mathis AG, 1992)	Only fabric					
Package dyeing (Fong's International, China, 2006)	Only liquor					
Jet (1992, Texam Limited, Nagaya Japan)	Liquor + Fabric					

2.3 Testing

K/S of all the samples was measured on a Datacolor reflectance spectrophotometer (Spectroflash-SF650X) under D65/10° illuminate. Percentage reflectance values at maximum wavelength (λ_{max}) are recorded against each sample for the result analysis¹⁶. K/S was calculated by using Kubalka Munk theory according to Eq. (1),

$$K/S = [(1-R)^2 / 2R] \quad (1)$$

Where S is scattering, K is absorbance and R is the reflectance. While K/S curve against wavelength is different for the different colours and their characteristics.

Colour fastness to washing was determined according to ISO 105-C06 A2S:2010 on Gyrowash (James H. Heel, Halifax England). Colour fastness to rubbing was measured according to ISO 105-X12:2002 on crockmeter (James H. Heel, Halifax England). ISO 13934-1:2013 test method for tensile testing using universal tensile strength tester (Lloyd instruments) was followed to measure the impact of dyeing on strength of the fabrics. Air permeability of the fabrics on SDL ATLAS M021A was measured following ISO 9237:1995. Scanning electron microscopy (FEI, Quanta 250) was used to analyse the structural changes after dyeing. One-way ANOVA was used to check the statistical significance of the results.

3 Results and discussion

3.1 Preliminary work

The dyed fabric samples at different time intervals were tested for K/S. The results showed that there is no increase in K/S value at 590 nm for Sample A to Sample E at 95 °C. The increase in temperature from 95 °C to 110 °C results in slight increase in the K/S as well as the reflectance wavelength at 590 nm due to the dye uptake of substrate as shown in Figure 2. Start of dyeing at 95°C is related to glass transition temperature of PET. The glass transition temperature (T_g) of PET is 85°C as the structure is dense and crystalline below this temperature so penetration of dye molecules at this temperature is almost impossible. The increase in temperature above T_g will promote the molecular chain mobility which facilitates the penetration of dye molecules. Hence the increase in temperature beyond 95°C promotes diffusion of dyeing molecules into the structure of PET and the effect is more obvious beyond 110°C and levels at 130°C (30 min).^{17,18} The increase in dyestuff concentration from 0.1% to 4% owf results in increased K/S and any further increase (4-10%) causes only slight increase in K/S as shown in Figure 3. Hence, 4% owf was selected for further experiments. The dyeing profile for all further work, Figure 1, was finalised based on preliminary results. The SEM images of the saturated dyed sample are given in Figure 4.

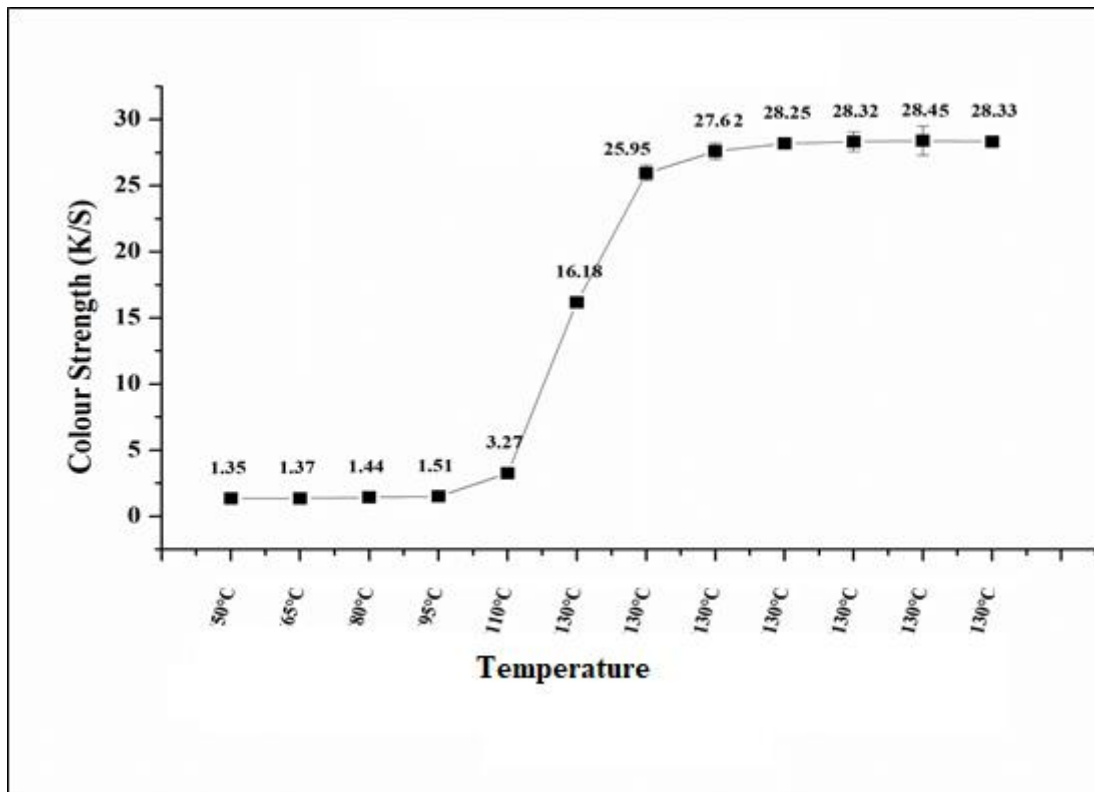


Figure. 2 Effect of dyeing temperature and time on K/S

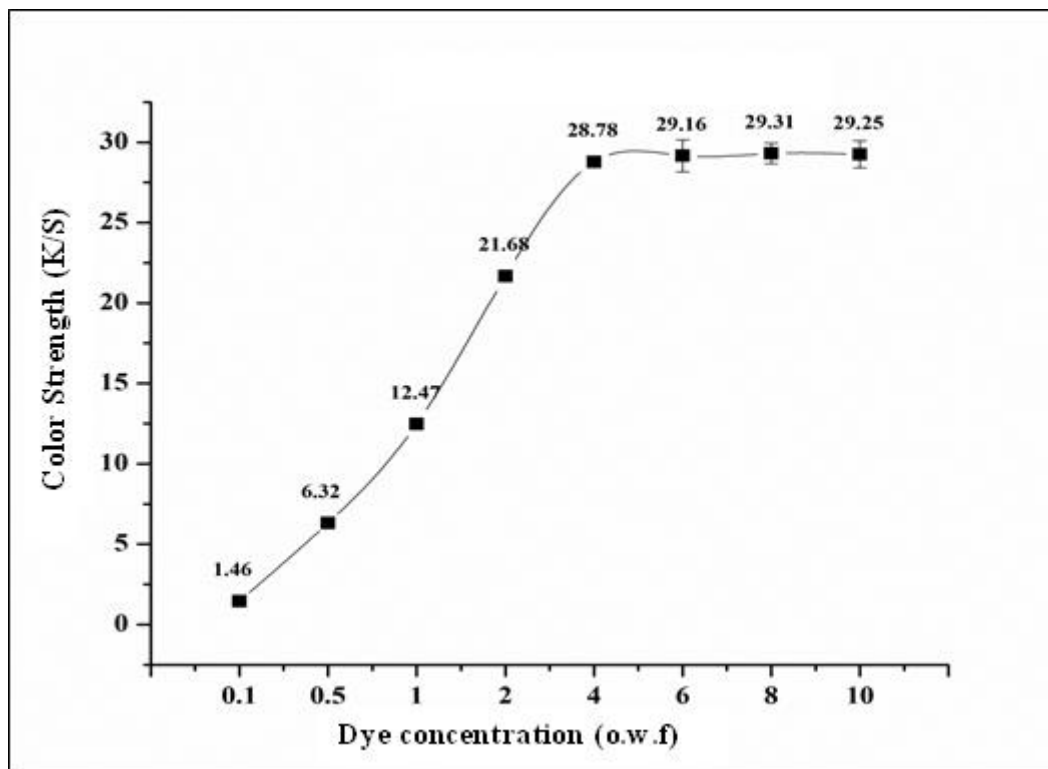


Figure. 3 Effect of increase in shade % on K/S

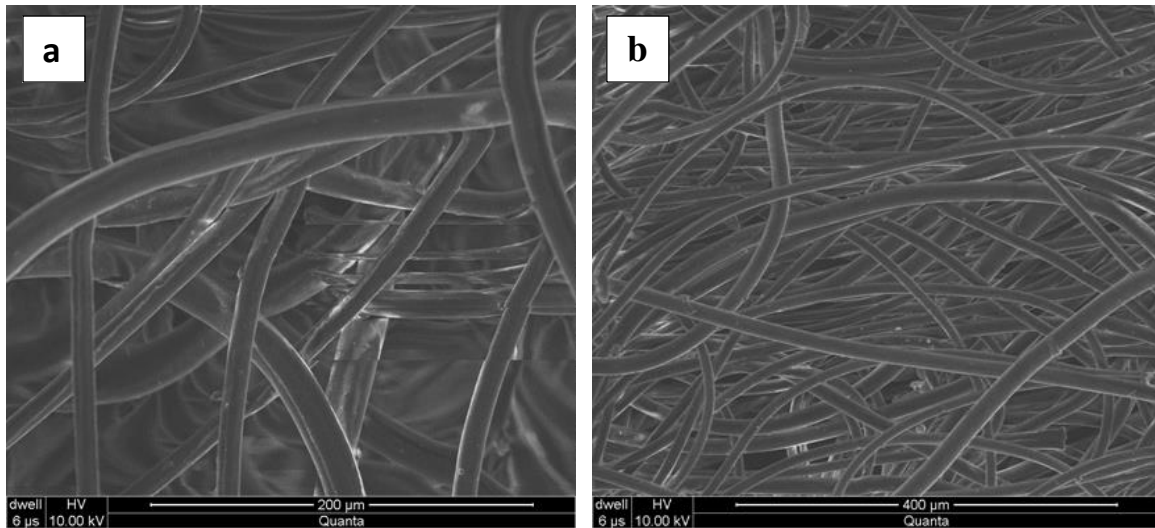


Figure. 4 SEM images of the saturated dyed samples

3.2 *Effect of linear density of PET fibers in nonwoven on K/S*

The results in Figure 5 show that the maximum K/S value is 29.61 for 100% 3 denier fibers in 200 gsm while least value of K/S is 23.96 with 75/25 blends of different denier fibers. Results in Figure 5 showed that K/S decreased with the increase of fine linear density (1.5 denier) fibre in the nonwoven fabric. The is due to the effect that for a given dye concentration produces a lesser depth of shade when present on a finer denier fiber ¹⁹. Since the amount of finer denier fibers in 75/25 blended fabric is greater than courser denier fiber so its shade depth is decreased as shown in Figure 5.

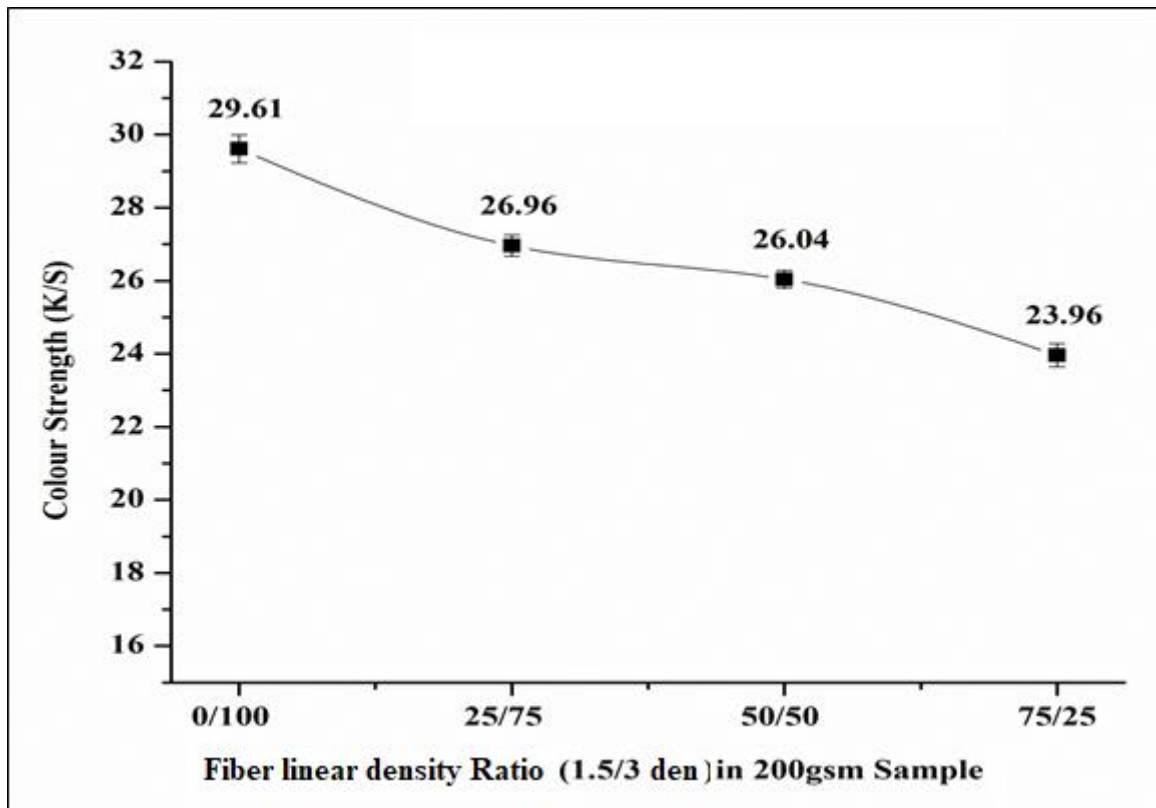


Figure. 5 Effect of fibre linear density on K/S value

3.3 Effect of Mass Ariel Density of nonwovens on K/S

The results in Figure 6 show that K/S value increases slightly with the increase in mass areal density of the nonwoven fabric. This slight, but significant, increase in K/S can be attributed to the increased K/S value (light absorbance by dyes) due to increase in the thickness of the fabric. Increase in GSM of fabric having same fiber linear density increases the number of chains which will enhance the amorphous region on the surface of the resultant fabric. Therefore, dye uptake was also increased with the increase of GSM due to the availability more amorphous region on the surface as shown in Figure 6.

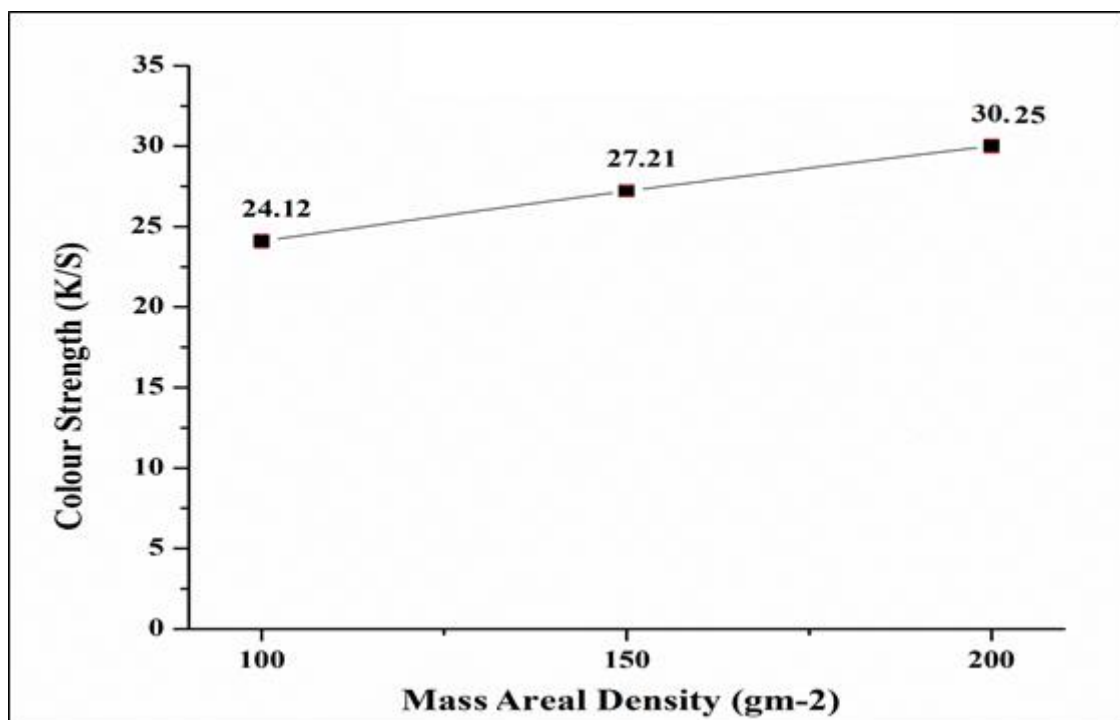


Figure. 6 Effect of mass areal density on K/S of nonwoven fabric

3.4 Comparison of dyeing methods

The samples dyed by different industrially relevant methods (Table 3) were tested for fabric performance. The K/S of Jigger and Package dyed samples was higher compared to Jet dyed samples, Figure 7. This can be attributed to the maximum interaction of the fabric with dye bath in both machine configurations, Figure 7, as fabric remains continuously dipped in the dye solution which facilitates the maximum dye migration from solution to substrate and results in higher K/S values. In jet dyeing, dyebath liquid pushes the fabric for the movement and both agitate in the whole process, while in package dyeing, the fabric is continuously in contact with the dyebath and dye bath liquid is pumped through the package with high pressure, which increases the rate of dye uptake in both dyeing methods. However, in jigger dyeing, dyebath liquor is stationary and fabric passes through it from one end to the second side by passing from the dye bath under tension of side roller, Figure 8. Hence the K/S of the Jigger dyed fabric is less, compared to jet and package dyeing, due to lesser residence time (and consequently less opportunity for the dyestuff to migrate) of the fabric in the dye bath.

The mechanical behaviour of needle-punched nonwovens in response to applied load produced higher extensions at smaller load which is likely to affect the fabric structure. The higher tension at high temperature and in the presence of water can affect the fabric structure as water can act as a lubricant and can facilitate fibre-fibre movement in friction-held nonwoven fabric. The deformed shape of Jigger dyed sample is shown in Figure 8.

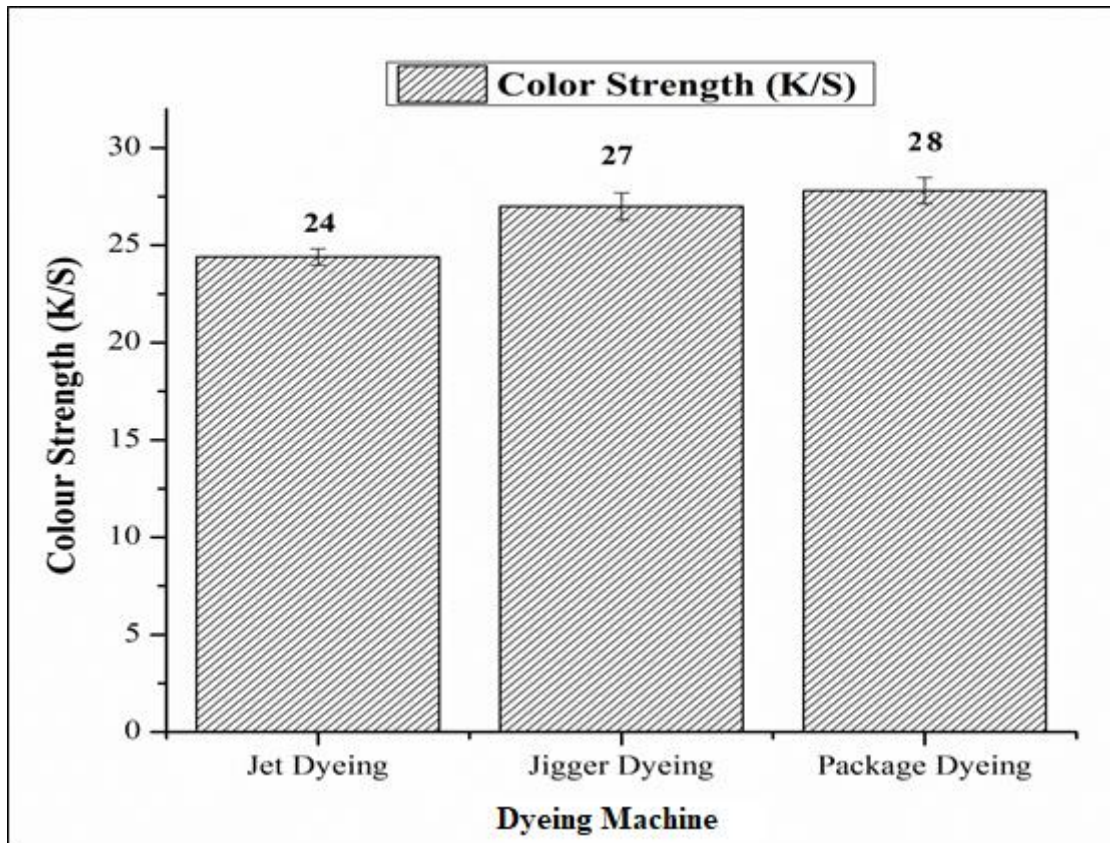


Figure. 7 K/S results of exhaust dyeing methods



Figure.8 Deformed jigger dyed sample

The colour fastness to washing and rubbing of all the samples graded 4/5 (excellent) in all types of conditions. Furthermore, post-testing (ISO 12945-2) no pills was observed on the fabric surface.

The tensile strength, Figure 9, of samples show characteristic machine- to cross-direction (MD:CD) ratio of cross-laid nonwoven fabric, where fibres are predominantly oriented in the cross-direction. MD:CD ratio of greige, Jet, Jigger and Package dyed samples was 0.44, 0.55, 0.19 and 0.49, respectively. The possible reasons can be the reorientation of fibre segments in response to applied mechanical forces under aqueous conditions. The strength of Jet and Package dyed samples increased in both MD and CD after dyeing as the extended exposure to high temperature can lead to compactness of the fabric ²⁰. The mass areal density of the fabric increased for package and jet dyeing upto 7% i-e from 100 gm⁻² to 107 gm⁻². In jigger dyed sample, the mass areal density of the sample was increased 15% (115 gm⁻²) from the original fabric mass areal density. As evident by Figure 8, the jigger dyed samples shrunk 52.5% from its original width. It can be possibly associated to the distortion of fabric structure due to the applied tension of take-up and let-off rollers. Under the applied tension, fabric starts to integrate in longitudinal direction. Hence, the tensile strength in cross direction is increased due to the integration of fibres from cross direction to machine direction and is decreased in the machine direction vice versa. The Package dyeing method can be preferred method for dyeing needle-punched (and other mechanically bonded nonwovens) due to least changed in MD:CD ratio compared to that of greige sample.

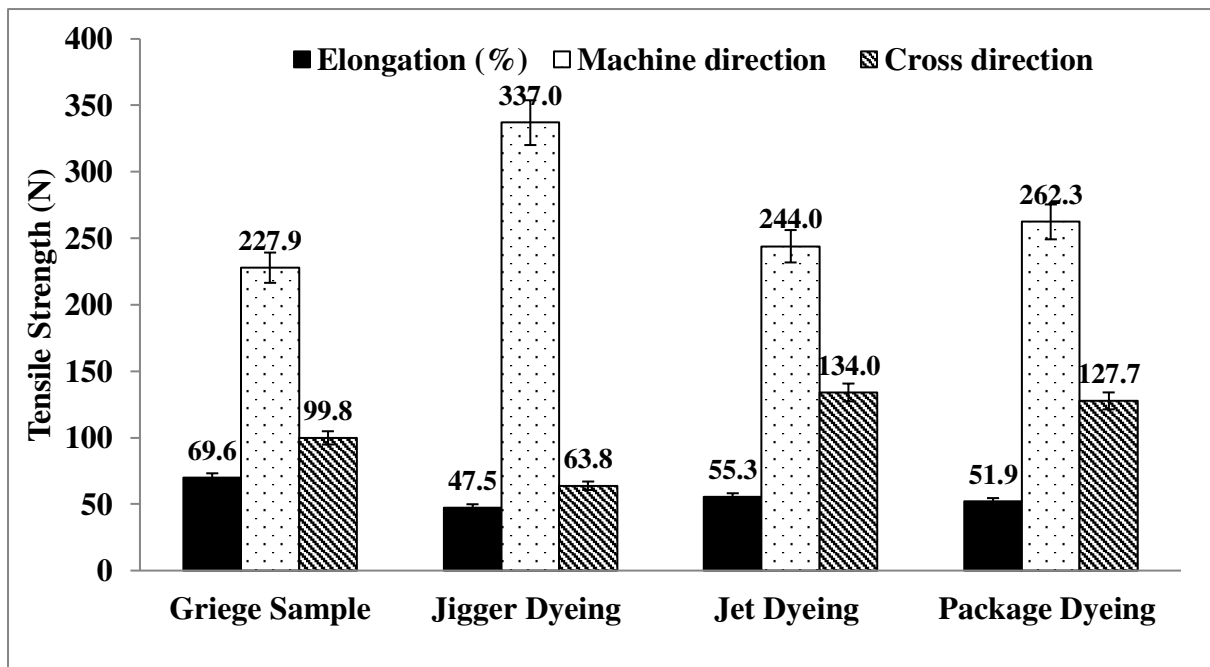


Figure.9 Comparison of tensile strength in the Machine and Cross direction

The air permeability of all the samples decreased post-dyeing as shown in Figure 10. The compactness of structure due to rearrangements lead to more resistance to air in all the samples and the effect is aggravated in case of Jigger dyes samples due to high fabric structural deformations (Figure 8).

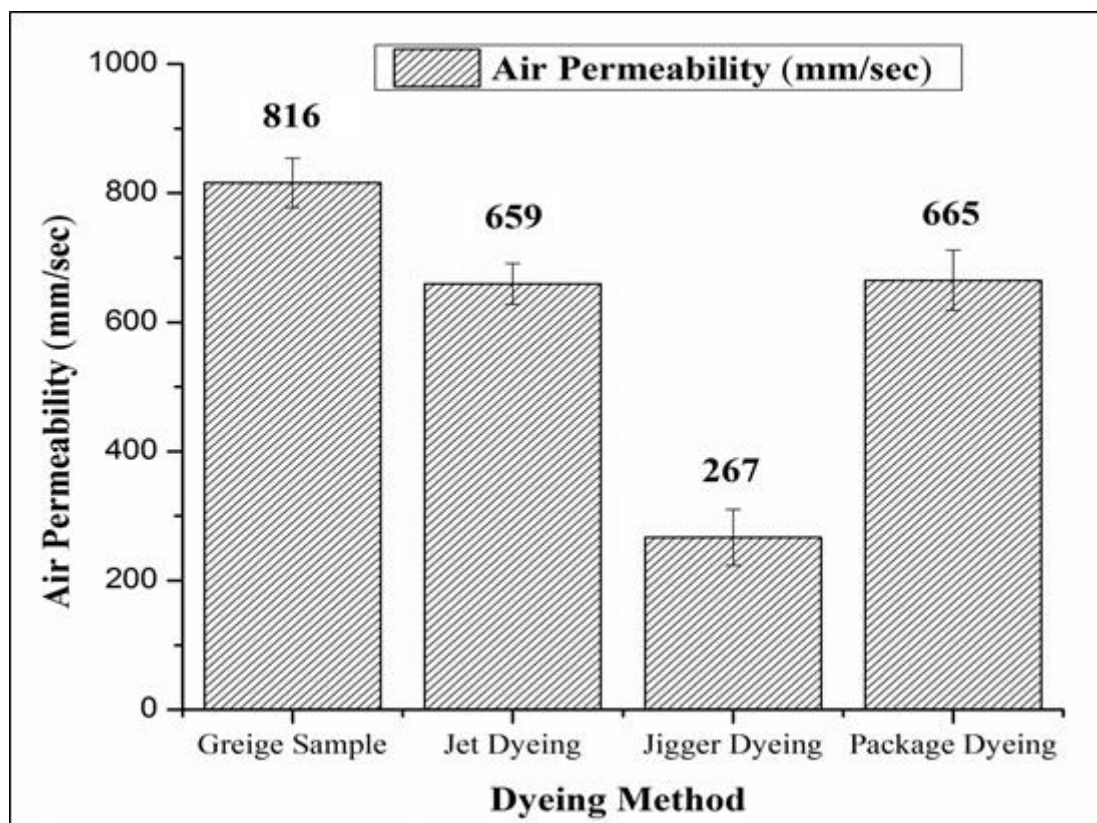


Figure. 10 Effect of different dyeing methods on Air Permeability

Conclusion

The dye saturation conditions for the dyeing of needled PET nonwoven fabric for higher K/S values are 4% owf, 130 °C for 30 min. The decrease in fibre linear density exhibits decrease in K/S of dyed needle-punched nonwovens because of the increase in surface area of the fabric. At same % owf, increase in PET nonwoven fabric areal density resulted in slight increase in the K/S. The comparison of industrially relevant dyeing methods showed that Jet dyeing and Package dyeing methods were more favourable for dyeing of needle-punched nonwovens whereas Jigger dyeing deteriorates the structural integrity of the fabric. All dyeing methods in this work resulted in reorientation of fibre segments, as shown by MD:CD tensile strength ratio of samples. However, package dyeing method showed the least change to fabric structure, as MD:CD ratio (0.49) was closest to the greige sample's ratio (0.44). All samples exhibited excellent colour fastness to washing and rubbing.

References

1. Tausif M, Goswami P. Nonwoven Fabrics. In: Cassidy T, Goswami P, eds. *Textile and Clothing Design Technology*. CRS Press; 2017:259-280.
2. Ahmad AI. Handbook of nonwovens. In: Russell SJ, ed. *Nonwovens Fabric Finishing*. woodhead publishing limited Cambridge England.
3. Wilson A. Development of the nonwovens industry. In: Russell SJ, ed. *Handbook of Nonwovens*. CRS Press; 2006:1-10.
4. Hussain T, Ullah A, Umar M, et al. Modelling the properties of pigment-printed polypropylene nonwoven fabric using the Box-Behnken technique. *Color Technol*. 2015;131(6):474-480. doi:10.1111/cote.12178
5. Waring, David R and Hallas G. *The Chemistry and Application of Dyes*. Springer Science & Business Media; 2013.
6. Aspland JR. Textile color application processes. *Color Res Appl*. 1983;8(4):205-214. doi:<https://doi.org/10.1002/col.5080080403>
7. Shakoor A, Baig GA, Tausif M, Gilani SQZ. Pigment and disperse printing of needlepunched polyethylene terephthalate nonwovens. *Dye Pigment*. 2017;136:865-872. doi:10.1016/j.dyepig.2016.09.052
8. Xu W, Yang C. Hydrolysis and dyeing of polyester fabric using microwave irradiation. *Color Technol*. 2002;118(5):211-214. doi:10.1111/j.1478-4408.2002.tb00101.x
9. Burkinshaw SM, Hewitt AD, Blackburn RS, Russell SJ. The dyeing of nonwoven fabrics part 1 : Initial studies. *Dye Pigment*. 2011:1-7. doi:10.1016/j.dyepig.2011.09.011
10. De Meyer T, Steyaert I, Hemelsoet K, Hoogenboom R, Van Speybroeck V, De Clerck

- K. Halochromic properties of sulfonphthaleine dyes in a textile environment: The influence of substituents. *Dye Pigment*. 2016;124:249-257.
doi:<https://doi.org/10.1016/j.dyepig.2015.09.007>
11. Canbolat S, Kilinc M, Kut D. The Investigation of the Effects of Plasma Treatment on the Dyeing Properties of Polyester/Viscose Nonwoven Fabrics. *Procedia - Soc Behav Sci*. 2015;195:2143-2151. doi:<https://doi.org/10.1016/j.sbspro.2015.06.278>
 12. Hoque MT, Mahltig B. Realisation of polyester fabrics with low transmission for ultraviolet light. *Color Technol*. 2020;(February):1-10. doi:10.1111/cote.12470
 13. Yi Z, Jihong F, Shuilin C. Dyeing of polyester using micro-encapsulated disperse dyes in the absence of auxiliaries. *Color Technol*. 2005;121(2):76-80. doi:10.1111/j.1478-4408.2005.tb00255.x
 14. Ferus-Comelo M. A new method to measure the solubility of disperse dyes in water at high temperature. *Color Technol*. 2015;131(4):269-271. doi:10.1111/cote.12153
 15. Shinde T, Marathe R, Dorugade VA. Effect of water hardness on reactive dyeing of cotton. *Int J Text Eng Process*. 2015;1(4):28-34.
 16. Mine Akgun, Behcet Becerir HRA. Assessment of color strength and color difference values of polyester fabrics containing continuous weft yarns after abrasion. *Fibers Polym*. 2007;8(5):495-500.
 17. Hawkyard C. Dyeing of Polyester Fibres. In: *Synthetic Fibre Dyeing*. Society of Dyers and Colourists; 2004:58-66.
 18. Park KH, Koncar V. Diffusion of disperse dyes into supermicrofibres. *Autex Res J*. 2004;4(1):45-51.
 19. Shin J, Bide M. Dye distribution in the dyeing of mixed denier polyester fabrics. *Color Technol*. 2000;116(10):305-309. doi:<https://doi.org/10.1111/j.1478->

4408.2000.tb00006.x

20. Nwachukwu AN IC. Effects of Time of Heat Setting and Wet Processes on Tensile properties of Griega Knitted Ingeo™ Poly Lactic Acid (PLA) Fabric. *J Text Sci Eng.* 2013;03(03):4-9. doi:10.4172/2165-8064.1000137