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CHARACTERISATION OF VANILLIN AND ITS APPLICATION AS CARRIER ON COMMERCIAL AZO DISPERSE DYES ON WOVEN POLYESTER FABRIC

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ABSTRACT

An extracted vanillin from Foster Clark vanilla flavour for the chemical substitution of toxic carriers used in aqueous dyeing of polyester fabrics was assessed. The extracted vanillin was used to compare the dyeing of a woven polyester fabric with two commercial dyes C.I Disperse Yellow 56, and C.I. Disperse Yellow 50. The vanillin carrier is used for dyeing at 3 % shade at boil, the dye uptakes increased for both C,I Disperse Yellow 56 and C.I Foron Yellow 50 , while higher at 0.15 g of vanillin at pH 4 gave 70.4 % exhaustion on C.I Foron Yellow 50 and least on C.I Disperse Yellow 56 gave 0.66 % at pH 11. The wash, heat pressing and light fastness was determined, an excellent wash fastness was observed at both ISO 2 and ISO 3 wash fastness respectively, given an excellent rating at 4-5. More so, the light fastness test gives a high fastness rating at an average of 6-7 for both the two commercial dyes at different pH and concentration used. The study confirms that vanillin can be used as a chemical substitute to conventional carriers using the aforementioned dyes and gave good wash and light fastness properties.

Keywords: *Vanillin, Polyester, Dyeing, Light Fastness, Wash Fastness*

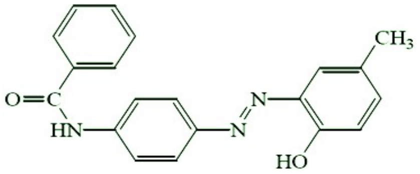
INTRODUCTION

Polyester fibres are difficult to dye with many disperse azo dyes because of the low rate of dye sorption at temperatures lower than the glass transition temperature (T_g=333-353 K); however, the presence in the dyebath of certain low-molecular-weight compounds (carriers), which are rapidly adsorbed by the fibre, markedly accelerates the dyeing rate.Carriers are used for dyeing of PET fibres in order to improve adsorption and accelerate diffusion of disperse dyes into the fibre at low temperature and pressure conditions. Nevertheless, most of carriers are toxic for humans and aquatic organisms (Murray and Mortimer, 1971; Yang and Huda, 2003). During dyeing and rinsing, a large amount of carriers is released into wastewater, but part remains entrapped in the fibre (Vigo, 1994) and is likely to be emitted into air during drying, thermo fixation and later use (e.g. ironing). Chemical carriers include: phenolics, chlorinated aromatics, aromatic hydrocarbons and ethers (Vigo, 1994). Some carriers are said "hydrophobic" and some are "hydrophilic", and their mode of action differ accordingly. Hydrophobic carriers are more effective than hydrophilic ones (Burkinshaw, 1995). In textile industry, hydrophobic carriers such as dichloro and trichlorobenzene are already substituted by hydrophilic carriers such as benzoic acid (Vigo, 1994).

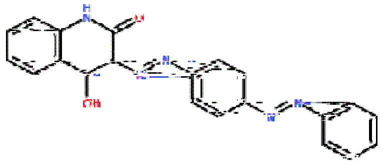
Molecular structure of vanillin is similar to that of traditional carriers, which confers to all of them a solubility parameter close to that of PET. Hence it would be interesting to study the possible use of vanillin to substitute traditional toxic carriers. Naturally occurring vanillin in pods is very expensive and was for a long time replaced by petrochemical vanillin for its use in poultry food and perfumery. There is now a great concern for its production using biotechnological solution: Rhodia markets biosynthetic vanillin prepared by the action of microorganisms on ferulic acid extracted from rice bran and today lots of research is being undertaken to synthesize vanillin from agro-resources such as lignin (McShan, 2005). Moreover, vanillin is antioxidant (Tai *et al.,* 2011) antimicrobial and anti-mutagenic effects (Walton *et al.,* 2003). But here attention is given to its extraction using vanilla flavour due to limited available resources.

Azo dyes are defined as compounds containing at least one azo group attached to sp^2 hybridized carbon atoms, such as benzene, naphthalene, thiazole and thiophene. Under normal conditions, azo dyes exist in the more stable planar transform so that the carbon nitrogen bond angle is approximately 120°. Azo dyes represent the largest group of disperse dyes for two reasons:

The ease with which an extraordinary number of molecular combinations can be generated by varying the diazo and coupling component. The relatively simple process by which the dyes can be produced. Colours that are less typical of simple azo compounds, such as greenish-yellow and blue are also possible using more specialized components. These may have heterocyclic units or cyano substituents.



C.I Foron Yellow 50



C.I Disperse Yellow 56

Fig. 1 Chemical structures of the azo dye used.

MATERIALS AND METHOD

Materials

The chemicals used for this work were of analytical grade mostly from BDH, May and Baker. These chemicals were used without further purification. Melting points were determined by the open capillary method. IR spectra were recorded using Perkin–Elmer model 881 spectrophotometer in the range of 4000–400 cm^{-1}. The chemicals and polyester fabric was purchased from Hadiss chemical stores Ltd., Samaru Zaria, Nigeria and Cardinal stores and chemical Ltd. Samaru Zaria, Nigeria and the fabric was purchased at the Samaru market Zaria Nigeria. C.I. Disperse yellow 56, C.I. Disperse Foron Yellow 50 purchased at Cardinal and chemical stores Ltd Samaru Zaria, Nigeria. The C.I. Foron Yellow 50 with a melting point of >360 °C, molecular weight of 331.37 g/mol, Ultraviolet–Visible (UV–VIS) Spectroscopic Analysis at 421nm, while the C.I. Disperse Yellow 56 with a molecular weight 369.38g/mol, Ultraviolet–Visible (UV–VIS) Spectroscopic Analysis at 437nm and melting point at >360 °C.

EXPERIMENTS

Extraction of Vanillin

500 mls of artificial vanilla chips flavour from Foster Clark was transfer into a 250mls separating funnel. This was then extracted using 120 mls x 4 of diethyl ether. The solution was shaken vigorously for several minutes, and then the layers were laid to settle down to separate. The aqueous layer was separated into a beaker, the ether extraction was kept in a round bottom flask. A support distillation panel was set to remove and recover the diethyl ether.

At the end of the distillation a yellow colour substance was obtained which was transferred into a beaker. The oil was allowed to settle at room temperature and then it solidified into a yellowish crystal. This was then allowed to rycrystallise below 80°C. (Nunn, 1979). The crystal was then allowed to dry over night and the melting point was determined using Gallenkamp apparatus.

Dyeing

The samples weighing 1g were dyed in 50ml beakers. With two different commercial dyes, (C.I Foron Yellow 50 and C.I Disperse Yellow 56). For each dyeing the liquor volume was set to 100ml. The amount of each dye used was 3% o.w.f at a liquor ratio of 50:1. The dyeing was carried out at a boil for 1 hour at different pH values (4, 5, 7, 9 &11) this was adjusted using aqueous potassium hydroxide and hydrochloric acid accordingly. Then, dyed samples undergo a reduction clearing using soda and sodium hydrosulfite for 30 min at 50°C, to remove all physi-sorbed dye molecules on PET fabric surface. At the end, dyed samples were washed twice at 30°C for 10 minutes in distilled water and dried at room temperature (Nkeonye, 1987). Dyeing was carried out using vanillin as carrier.

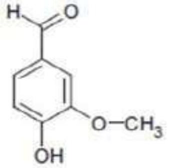


Fig. **2: Vanillin structure**

Dyed samples were tested according to ISO standard methods. Specific tests used were ISO wash fastness No. 2 and 3 respectively. For the fastness to washing, a specimen of dyed polyester fabric was washed at 50°C for 45 minutes. Colour change was evaluated by comparison with a non-washed specimen. Colour difference was measured to assess the wash colour fastness: 1- poor, 2 - fair, 3 - moderate, 4 - good, 5 - excellent. Also the fastness to pressing was also done at dry, damp and wet to assess the use of heat on the fabrics.

RESULTS AND DISCUSSION

The melting point of the extracted vanillin was found to be at 178-179^{0}C, yield obtained was about 4.65g, and it also showed an absorption at 3173cm^{-1} due to OH stretch vibration, 1154-1200cm^{-1} due to C-H stretch aromatic vibration, 1300cm^{-1} due to C-H stretch aliphatic, 1509-1558cm^{-1} due to C=C stretch, 1028cm^{-1} due to CO stretch, and 1772 due to C=O stretch and finally, 812.56 due to C-C stretch respectively.

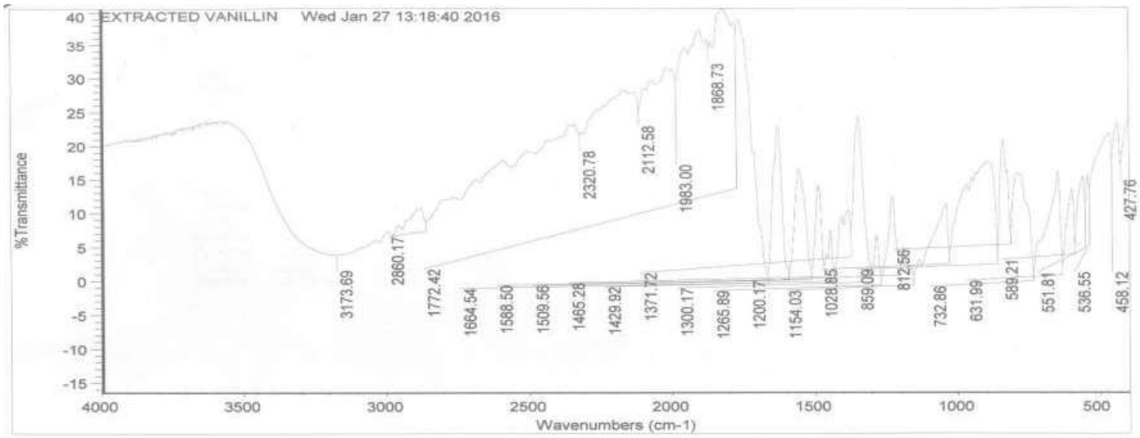


Fig. 3: Infrared spectrum of the extracted vanillin

Table 1. Percentage Exhaustion of Dyes on Polyester Samples Using Vanillin

pH	**0.05g**		**0.10g**		**0.15g**	
	D.Y	F.Y	D.Y	F.Y	D.Y	F.Y
4	11.8	62.5	12.5	69.5	12.5	70.4
5	7.9	60.9	8.3	63.6	9.0	65.5
7	4.9	46.7	5.0	54.3	5.6	57.8
9	1.98	16	0.66	37.5	0.7	35.6
11	0.66	17	3.5	42.6	3.7	62.4

where: D.Y = C.I Disperse yellow 56, F.Y.= C.I. Foron Yellow 50,

Table 2. Exhaustion (%) of Vanillin on Commercial Dye without pH Control

Concentration (g)	**C.I FORON YELLOW 50**	**C.I DISPERSE YELLOW 56**
0.05	61.2	19
0.10	63.6	7.4
0.15	69.1	12.1

Table 1 shows the effect of pH on the dyeing exhaustion of the dyes respectively. It was observed that there was an increased in dyeing exhaustion at pH 4 to pH 7 and then subsequent decrease at basic pH value 9 to11 for all the dyes used. This results shows that at high pH values, electrostatic repulsion between negatively charged PET, and negatively charged dye and vanillin, would make dye adsorption more difficult and would thus decrease the dye uptake. (Ran *et al.,* 2011). Also (Nunn, 1979), affirmed that in an acidic condition less than 4 or in an alkaline bath, more rapid will occur which can induce serious degradation of polyester fibres, therefore the bond of attachment of the fibre and the dye is weak for dye adsorption and absorption.The results shows a higher percentage exhaustion when the C.I Foron Yellow 56 was used than the C.I Dispersed Yellow 50, The variation in exhaustion could be as a result of the closeness to the solubility of the C.I Foron Yellow 50, than the C.I Disperse Yellow 56 and. This was affirmed by (Walton *et al.,* 2003): "Dye having the solubility parameter close to that of the polymer would have high exhaustion". This results proves that the effect of pH on dye exhaustion is greatly dependent on the nature of the dye and the pH used (Walton *et al.,* 2003).

Table 3. ISO2 Wash Fastness of the Commercial Dyes Using Vanillin

	0.05g				**0.10g**				**0.15g**			
pH	FY		DY		FY		DY		FY		DY	
	CS	S	CS	S	CS	S	CS	S	CS	S	CS	S
4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
5	4-5	4-5	5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
7	4-5	4-5	5	4-5	4	4	5	5	4	4-5	5	5
9	4-5	4-5	5	5	4-5	4-5	5	5	4	4	4-5	4-5
11	5	5	5	5	4-5	5	5	5	4-5	4-5	4-5	4-5

Table 4. ISO 3 Wash Fastness of the Commercial Dyes Using Vanillin

	0.05g				**0.10g**				**0.15g**			
	FY		DY		FY		DY		FY		DY	
pH	CS	S	CS	S	CS	S	CS	S	CS	S	CS	S
4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5	4-5	4-5	4-5	4-5
5	4-5	5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
7	4-5	4-5	4-5	4-5	4	5	4-5	5	4-5	4-5	4-5	4-5
9	4-5	4-5	4-5	4-5	4-5	5	4-5	5	4-5	4-5	4-5	4-5
11	4-5	5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5

The ratings vary from good to very good for the dye at 0.05, 0.1 and 0.15g vanillin, at the given pH of 4, 7, and 9, which shows a very good result. While the ISO No.2 wash fastness for the disperse dye shows an excellent result at 0.05 under the same pH condition. The fastness results obtained are almost the same, whatever the pH used. This excellent results could be as a result of the size and polarity of the dye molecule which reduces it mobility within the fibre and be reflected in a lower diffusion coefficient and an attendant increase in wash fastness because polyester is highly crystalline and hydrophobic. Both properties restricts diffusion. The former directly, by presenting a close packed structure as an obstacle to movement, and the later by reduced fibre swelling which serves to keep the structure tightly packed.

There is generally good light fastness for the two dyes. This results could be as a result inherent photo-stability of the azo dye molecules, the concentration of the dye within the fibre, the wavelength distribution of the incident radiation and the composition of the atmosphere (Giles, 1974). Also the Dye contain amino group and hydroxyl group which are hydrogen bonded onto the azo linkage. The double hydrogen bonding prevents the tautomerism that is normally feature of naphthalene based azo dyes by locking the dye in the azo form, leading to the high level of light fastness ratings couple with the presence of NO_2, and OCH_3 in the dyes. The good light fastness could also be attributed to the molecular structure of the dye, planarity and dye substituent that provided shield from radiant energy on azo chromophores.

Table 7 shows the results to fastness to pressing on the polyester. The results shows that the commercial dyes had good to very fastness to pressing.

Pressing fastness can be an important property of disperse-dye on polyester, because of the use of heat treatments in the finishing of the fabric. Disperse dyes are not chemically combined with the fibre or fabric, but are mechanically held in solid solution. Thus, when they undergo the influence of heat they are good to vaporise out of the fibre with little or no decomposition (Hauser, 2011).

Table 5. Light Fastness of Dyed Samples Using Vanillin

pH	**0.05**		**0.10**		**0.15**	
	FY	DY	FY	DY	FY	DY
4	7	7	7	7	6	7
5	7	7	7	5	6	7
7	6	5	6	6	7	6
9	7	6	7	6	6	6
11	6	6	7	6	6	6

Table 6. Light fastness for Neutral Vanillin

Foron Yellow 50		**Disperse Yellow 56**	
Grams (g)	Light Fading	Grams (g)	Light Fading
0.05	6	0.05	6
0.1	7	0.1	7
0.15	6	0.15	7

Table 7. Fastness to Heat Pressing of the Commercial Dyes

	DRY				**DAMP**				**WET**			
pH	FY		DY		FY		DY		FY		DY	
	CS	S	CS	S	CS	S	CS	S	CS	S	CS	S
4	4	4	4	1	4	4-5	4-5	5	4-5	4-5	4-5	4-5
5	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5	4	4-5	4-5	5
7	4-5	4-5	4	4	4	4	5	5	4-5	5	4	4-5
9	4-5	4-5	5	4	4-5	4-5	4	4-5	4	4-5	4-5	4-5
11	4	4-5	5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5

CONCLUSSION

This study was carried out to study the potential use of an extracted vanillin molecule for substituting conventional carriers and its application on two commercial azo dye (Disperse Yellow 56, and Foron Yellow 50) and on polyester fabric dyeing process. Best dye uptake was observed at acidic condition at pH 4. The study showed that vanillin as carrier allows a uniform distribution of the various disperse dyes in the polyester fibre. Thus dye exhaustion was high, there was an excellent wash/light fastness and heat pressing fastness. There was an increase in the dye uptake as the concentration of the vanillin increases. The study also confirms that dyeing with vanillin can be carried at neutral pH without addition of other chemicals to adjust pH. This result also confirms that the dye used in the study diffuses uniformly inside the PET fibre.

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