



Development of dissolvable dye transfer inhibitor for household laundry

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This research is focused on developing a dissolvable composite polymeric film loaded with a dye transfer inhibitor. Pyridine N-oxide (PNO) is one of the amine-based dye transfer inhibitor (DTI) polymers with higher dye inhibition ability. A transparent and water-soluble PNO-loaded polymeric composite film of polyvinyl alcohol (PVA-PNO film) has been developed using the solution casting method, and the process parameters are optimised. The performance of the developed polymeric composite film has been evaluated in terms of dissolving time and dye-transfer inhibition in the laundry. The results indicate that the addition of the cross-linking agent increases the dissolving time of the film, while the addition of the plasticizer reduces it. An average of 8.5 min is observed as the dissolving time for the developed product in the presence of detergent. In the laundry process, a colour difference value (ΔE) of 4.763 is noted against Reactive Blue 4 ($C_{23}H_{14}Cl_2N_6O_8S$), and the performance of the product against multiple dyes is found to be satisfactory. The tensile strength of the film is found as 17.1 Mpa, elongation 113%, and folding endurance 250 times, confirming the better-handling property of the developed film.

Keywords: Colour difference value, Dissolving time, Dye transfer inhibitor polymer, Pyridine N-oxide film, Polyvinyl alcohol film

1 Introduction

Dye transfer inhibitors (DTI) are used to prevent colour re-deposition in washing machine cycles, which allow coloured clothes to be washed together with white clothes without staining them. The problem is attributed to different factors related to the washing process and also based on the quality of the dyes and auxiliaries used. Several researchers have reported different polymers as dye transfer inhibitors, where the commonly used polymers are polyvinyl pyrrolidone (PVP), polyvinylimidazole (PVI), and polyamine-N-oxides such as polyvinylpyridine-N-oxide¹⁻³.

DTI polymers bind irreversibly and hence, the free dye molecules in the wash water form water-soluble macromolecules, thus preventing them from re-depositing onto garments; these macromolecules are finally removed with the water⁴. However, one of the major issues with the DTI polymer is the proper way of introducing the polymer into the wash cycle. Several researchers came up with various methods to introduce the dye transfer inhibitors into the wash liquor, washing compositions, softening compositions⁵⁻⁷, detergent compositions⁸⁻¹⁰, and as a separate laundry aid product with support matrix¹¹⁻¹⁵. Out of the

mentioned methods, the use of the support matrix is a widely adopted method due to its ease of use and better control over the quantity of the DTI polymer that can be introduced for every cycle of the wash. In 1999, Felstead and Ziskind¹⁴ filed a patent for dye scavenging articles made up of cellulose-based woven or non-woven material, where the DTI polymer was chemically bonded to the support matrix.

In the most adapted support matrix system, various structures were included to introduce more amounts of DTI polymers on the surface of the matrix. Another study¹⁶ found that the single-layer DTI articles were frequently affixed to the laundry substrate, and caused undesired effects. To control this issue, they developed a multi-layer dye-scavenging article, which prevents contact between the articles or garments. One of the important and most noted problems with the support matrix structure is the entrapment of DTI articles within the garments. Ziskind¹¹ developed a structure to prevent the article from becoming trapped and/or commingled with the clothing during the entire laundry washing cycle. Wattiez and Hennion¹⁷ developed a layer of the cellulosic barrier which was coated with DTI article to wash the different coloured garments in the same wash load. Felstead and Ziskind^{12,18} developed a support matrix with a flotation member, to avoid getting trapped in clothing,

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and a stiffening member to keep the article in the open state during washing. Panandiker *et al.*^{13,19,20} developed a multilayer article to disperse the DTI polymer with enough physical strength and stiffness throughout the washing cycle so that it can deliver the DTI polymer efficiently and remains in the open state during the whole washing. Orlandi *et al.*²¹ developed a support matrix-based system to solve multi-functional issues by introducing DTI and fabric softening components in the carrier matrix. However, the issues remain unsolved as every product has its limitations.

Polyvinyl alcohol (PVA) was synthesised by the polymerization of vinyl acetate to polyvinyl acetate (PVAc), which was then hydrolysed to get PVA²². The PVA has a hydroxyl group in its structure. The degree of polymerization (DP), hydrolysis, and solution temperature are the three factors that determine the solubility of PVA in water²³. As PVA is water-soluble, PVA films can be formed without the use of noxious solvents. PVA films are easily prepared by the evaporation of an aqueous solution of the polymer. The resultant films are resistant to tear, with a high degree of clarity and gloss, and do not irritate the skin²⁴. However, the water resistance of the PVA film is one of the main disadvantages. Lim and Wan²⁵ observed that the addition of glycerol or polyethylene glycol into PVA films increases film flexibility, thus lowering the water resistance and enhancing their aqueous solubility. Pyridine-N-oxide (PNO) is one of such well-known DTI polymers and it binds the free dye molecules in the wash water and forms water-soluble macromolecules². Studies reported that the performance of PNO was superior to PVP due to its aromatic structure²⁶. Hence, PNO was selected as a DTI polymer for the current study.

As far as the previous studies are concerned, most of the works were dedicated to analyse the type of support matrix material, the number of layers, and incorporation methods to avoid the article being trapped inside the wash loads. In all these methods, the unaddressed problem is the removal of support matrix manually. To the best of our knowledge, no literature was found on this particular issue. Hence, this research was focused on developing a dissolvable polymeric (PVA) composite film loaded with PNO as DTI polymer using the solution casting method. PVA films, known for their water-soluble properties, was already used in packaging^{27,28} and dissolvable pouches for laundry additives^{29,30}. Here, the composite film was developed using PVA as a film-forming

agent. The developed film was evaluated for its mechanical properties and solubility in water. The product was also evaluated for its performance against different dyes and their mixture, and the findings as colour differences were analysed.

2 Materials and Methods

2.1 Materials

The DTI polymer [Pyridine N-Oxide (PNO)] was procured from Sigma Aldrich, USA (PNO: molecular weight 95.10, Purity 95%). Reactive Blue 4 ($C_{23}H_{14}Cl_2N_6O_8S$, molecular weight 637.43, dye content 35%), Acid Red 1 ($C_{18}H_{13}N_3Na_2O_8S_2$, dye content 60 %, molecular weight 509.42), Basic Blue 3 ($C_{20}H_{26}ClN_3O$, dye content 25 %, molecular weight 359.89), Disperse Orange 3 ($O_2NC_6H_4N=NC_6H_4NH_2$, dye content 90 %, molecular weight 242.23), Direct Red 80 ($C_{45}H_{26}N_{10}Na_6O_{21}S_6$, dye content 25%, molecular weight 1373.07) were procured from Sigma Aldrich, USA. Polyvinyl alcohol (PVA) having the specifications and molecular formula $[CH_2CH(OH)]_n$, molecular weight 85000, 99% hydrolysed and a water-soluble synthetic polymer was used as a film-forming agent. Citric acid, a weak organic tri-carboxylic acid ($C_6H_8O_7$, 99% purity) was used as a cross-linking agent at different concentrations (by dissolving in water), and glycerol ($C_3H_8O_3$, 99% purity) was used as a plasticizer; these were sourced from Mahalakshmi Scientific Company, Coimbatore. The commercial detergents/soap powders used were obtained from retail outlets in Coimbatore and used as such. The bleached cotton fabric (140 EPI and 80 PPI) was sourced from commercial outlets in Coimbatore and washed before use. All the chemicals used in this research were of analytical grade and used as obtained from the manufacturers.

2.2 Methods

2.2.1 Composite Film Preparation

Homogenous PVA-PNO composite film was prepared by solution casting technique. Ten grams of PVA polymers, known percentages of cross-linking agents, and plasticizers are mixed with 100 mL water using a magnetic stirrer. The mixture was stirred for 15 min at 35°C to get a homogeneous solution. A 0.25 g/L of DTI polymer (PNO) was added slowly to the PVA mixture and stirred for a further 15 min. The homogeneous solution was poured into a petri dish and then kept in an oven at 50°C. After 6 h, the petri dish was taken out and the film was peeled off gently^{31,32}.

2.2.2 Performance Analysis of Developed Product

The initial trials were conducted to analyse the performance of selected DTI. About 3% of dye (on the weight of the material) was added to one litre of tap water and the solution was stirred well to ensure that the dye particles were dissolved homogeneously in the water. Commercial detergent powder (2-10g/L) was added to the solution. The developed polymeric composite film along with swatches of white fabrics was then immersed into the solution. The wash liquor bath was kept at room temperature (35°C) under continuous agitation for about 30-45 min, after which the samples were removed from the wash liquor and washed under running water and then left to air dry. Once the samples were completely dried their colour difference (ΔE) values were analysed using a spectrophotometer.

The test was performed with the developed composite film (PNO-loaded PVA film), with the direct addition of PNO and also PVA film without PNO to evaluate the required concentration of polymers, and to analyse the impact of detergent and pH ³³. In this process, once the dye was added to the water, the reactive dye started to hydrolyse and the subsequent additions of DTI polymer initiated interaction between the DTI and dye molecules through a possible intermolecular H-bonding. The -OH components in the hydrolysed dye molecules acted as an electron-donating medium and easily adhered to the surface of the DTI, resulting in an inactive dye molecule irrespective of the liquor pH .

In the case of the multiple dye interaction experiment, individual dyes (3% on the weight of the material) were hydrolysed separately and mixed in the wash liquid, and later the developed product was added into the dye mixture along with fabric, as mentioned earlier.

2.2.3 Mechanical Property Evaluation

Tensile testing was conducted on rectangular specimens of 50mm×150mm, using a tensile testing machine (Uni-stretch 250) fitted with a 100 N load cell according to ASTM D5035-06. The initial distance between the grips and the cross-head speed was kept constant at 50 mm and 10 mm/min respectively. The values of the tensile strength and elongation-at-break of the polymeric composite films were averaged over three readings, and the thickness of the films was measured as per the ASTM D1777-96, using a precision thickness gauge.

2.2.4 Folding Endurance

The developed composite film was tested for its flexibility by physically folding and recovering at the

same place till it tore off or folded up to 300 times manually. This test parallels the practical handling of the film during the laundry process. The number of times the film could be folded at the same place without breaking gave the value of folding endurance³⁴.

2.2.5 Colour Value Evaluation

The CIELAB colour values (L^* , a^* , b^* and ΔE) of the dyed fabric were determined by measuring their surface reflectance using a computer-aided Spectra Scan 5100 A spectrophotometer (Premier Colors can India Pvt Ltd) followed by calculating the K/S values using Kubelka Munk equation with the help of relevant software. The measurement range was 360-750 nm at an interval of 10 nm as per AATCC – Test method – 153 – 1985.

2.2.6 Dissolving Ability of Film

To analyse the solubility time requirement for the developed product, the film was allowed to dissolve in one litre of water in the presence of 5g of detergent (one scoop/litre) and stirred well using a magnetic stirrer, and the time required to dissolve the product was measured in seconds.

3 Results and Discussion

To analyse the effect of cross-linking agent, plasticizer, and DTI polymer additions on the performance of the PVA film, different concentrations of the chemicals are added individually and their performance is analysed in terms of their dissolving ability.

3.1 Effect of Citric Acid on Dissolving Time of Film

Citric acid is one of the poly-carboxylic acids with numerous advantages, namely inexpensive and non-toxic³⁵. It has already been approved by the FDA for use in edible items³⁶. Citric acid consists of one hydroxyl and three carboxyl groups. Due to this multi-carboxyl structure, citric acid is also used as a cross-linking agent in many industrial applications. The citric acid forms covalent bonds to bind the internal molecules of the polymer and reinforces the structure³⁷. Hence, the mechanical properties, like tensile strength, handling ability, and solubility of the films, could be altered based on the requirement. The previous researchers also pointed out that the addition of a higher amount of citric acid may result in an esterification reaction between the multi-carboxylic groups and the hydroxyl groups in the PVA and so the water resistivity of the film could be improved³⁸. The interaction mechanism as reported earlier³⁹, between PVA and the cross-linking agent is provided in Fig. 1 (a).

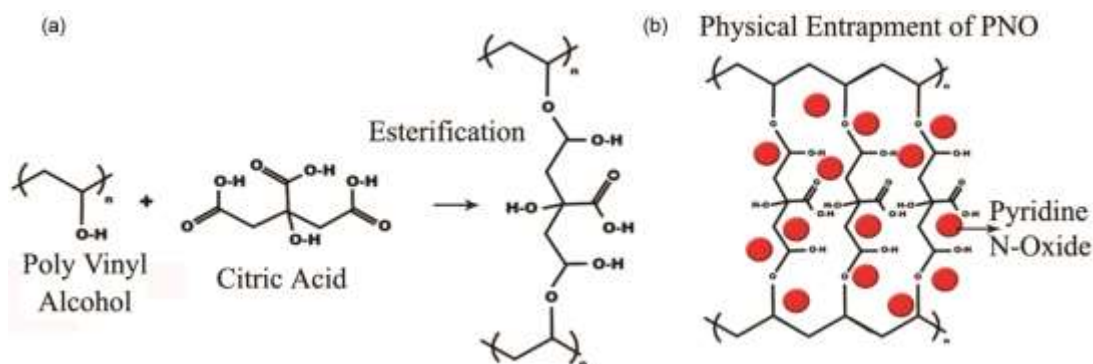


Fig. 1 — Interaction mechanism of PVA and citric acid (a), and illustration of physical entrapment of PNO in cross linked PVA film⁴²(b)

The PVA film has been prepared as a laundry additive because its quick dissolvability is the desirable characteristic. Though the film has an extremely low dissolving time, the lower strength of the film makes it very difficult to be used for the intended purpose. To improve these characteristics, a minimal addition of cross-linking agent is preferred. As the average washing cycle in a washing machine is up to 30 min, the desired dissolving time of the film needs to be not more than 10 min. In this aspect, various concentrations of citric acid (0-10 mL/100 mL of PVA polymeric solution) are added during the PVA film formation process and the dissolving ability is analysed. Figure 2(a) represents the dissolving time of the film in min. From the results, it can be observed that an increment in the concentration of the cross-linking agent increases the dissolving time of the film. The major reason for the increase is the formation of cross-links with the inter-molecules of the PVA and hence, the cross-linked structures are able to limit the movement of the molecular segments⁴⁰. This limited segment movement in the PVA film increases the brittleness and water resistivity of the PVA film. These results are found in line with the findings of Shi *et al.*⁴¹, who evaluated the influence of citric acid percentage on the structural changes of PVA and starch film. From the results, it can be noted that 1-2 mL of citric acid added to PVA films show less than 10 min of dissolving time. The result shows that 2 mL citric acid is the optimum value for composite film.

3.2 Effect of Glycerol on Dissolving Time of Film

Plasticizers are low molecular weight compounds, which are generally used along with polymers to improve the flexibility of the film. The addition of plasticizers like glycerol improves the handling properties of the film by reducing the brittleness of

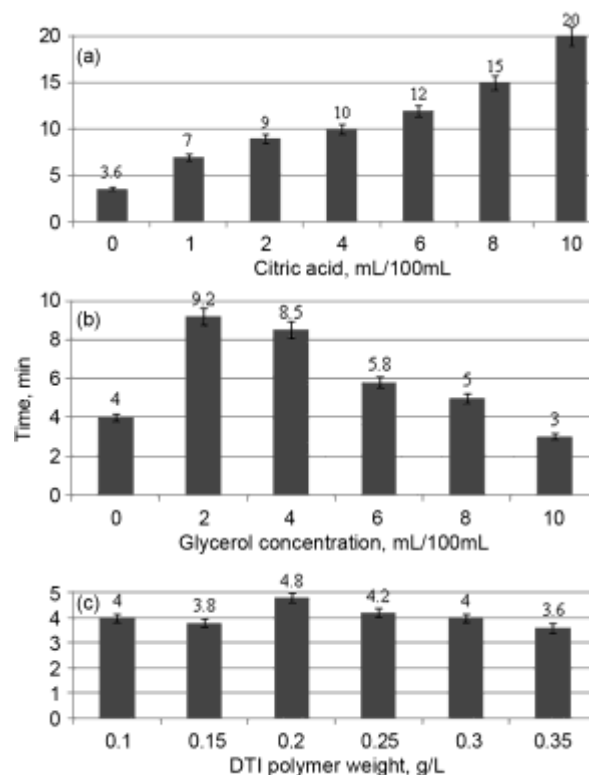


Fig. 2 — Effect of (a) citric acid concentration, (b) glycerol concentration and (c) DTI polymer concentration on the dissolving time of PVA film

the film as well as its flexibility and prevents it from cracking during enduse⁴². The selection and quantity of plasticizers are generally based on the application requirements and compatibility of the plasticizer with the polymer⁴³. In this study, to improve the handling and dissolvability of the film and also to reduce the brittleness, glycerol (1-10 mL per 100 mL of PVA polymeric solution) is added during the PVA film preparation process. Figure 2(b) represents the effect of glycerol concentration on the dissolving time of the film, considering 2 mL of citric acid addition

(optimum value). The reduction in the dissolving time is due to the addition of low molecular weight glycerol into the PVA polymeric structure. Glycerol increases the macromolecular mobility of the polymer, as well as the extensibility and flexibility of the film⁴⁴.

The plasticizer molecules in a polymeric system readily form hydrogen bonds with the PVA molecules. This cross-linking of PVA molecules with glycerol by hydrogen bonds hinders the formation of PVA crystallites in the plasticized films, as the size of the glycerol molecules is larger. Technically, the addition of glycerol forms smaller and probably more defective crystallites in its structure²⁵. When the plasticizer-added film is exposed to the water, the presence of glycerol in the PVA film increases the flexibility and transforms the PVA film into more permeable to water molecules, as the addition of glycerol reduces the number of crystallites and the quality of the crystallites in the PVA film. This phenomenon increases the solubility of the film with the addition of the plasticizer in the PVA film²⁵. However, the researchers also mentioned that the incorporation of a higher percentage of glycerol content in a PVA or starch film may create a phase separation among the solvent and result in sudden strength loss and increased solubility⁴⁵. Furthermore, a gradual decrease is observed in the solubility time, when started adding glycerol to the PVA film from 2 mL (a lower percentage) to 10 mL. The result obtained for the addition of 8 mL of glycerol per 100 mL of PVA solution, is found as the optimum value for this research. Further increase in glycerol concentration affects the handle properties of the film.

3.3 Effect of DTI Polymer on Dissolvability of PVA Film

The dye transfer inhibition property of Pyridine N-oxide (PNO) has been well studied and it is preferred due to its cost-effectiveness, non-toxicity and better performance owing to its chemical nature. In our previous work, the maximum required concentration of PNO per litre of wash liquid has been optimised statistically (0.25 g/L)²⁶. Based on the previous knowledge of the DTI polymer, the effect of different concentrations of DTI polymer on the dissolving ability of the film is evaluated. The polymer concentration is increased from 0.1 g (per 100 mL of PVA polymeric solution) to 0.35g. The results are depicted in Fig. 2(c). From the results, it can be understood that the addition of DTI polymer does not influence the dissolving ability of the PVA film.

These characteristics are attributed to the physical entrapment mechanism of DTI polymer [Fig. 1(b)] in the cross-linked PVA film⁴⁶⁻⁴⁸. Based on this analysis, the process parameters such as concentrations of DTI 0.25 g/L, cross-linking agent 2 mL/100 mL of PVA solution, and plasticizer of 8 mL/100 mL of PVA solution are taken for the PVA-PNO composite film preparation.

3.4 Product Characterization

3.4.1 Mechanical Property Analysis

Table 1 shows that the mechanical properties of the developed composite film are very much comparable with pure PVA film. The similar (like pure PVA film) tensile strength and elongation of the composite film represent that the developed film can withstand the tension and pressure applied during the practical handling process. Furthermore, the folding endurance test results are found promising that the film can endure up to 250 times of folding at the same place without tearing. These findings further support the stability of the film in the handling process. The developed film and pure PVA film, have a dissolving time in a similar range. It can be concluded that the addition of DTI polymer would not have any influence on the composite film, regarding the dissolving time.

3.4.2 Laundry Performance Evaluation of PNO Film

The results of the laundry simulation experiment are provided in Figs 3 and 4. The results are found similar to the direct addition of DTI polymer. The performance of the PVA-PNO composite film is also significant in terms of resisting colour re-deposition from the wash liquor. The visual assessment performed on samples [Figs 3(b) and (c)] shows no difference among them, whereas the control sample [Fig. 3(a)] used in the laundry without DTI polymer shows colour deposition. The samples were also analysed objectively to know the colour difference value (ΔE) by comparing them with a

Table 1 — Mechanical properties of PVA and PVA-PNO composite films

Film	Tensile strength Mpa	Elongation %	Thickness mm	Folding endurance No. of times	Dissolving time, s (min)
PVA film*	19.25	125	0.157	255	510 (8.5)
PVA-NO* composite film	17.1	113	0.162	252	514 (8.56)

*n=3.

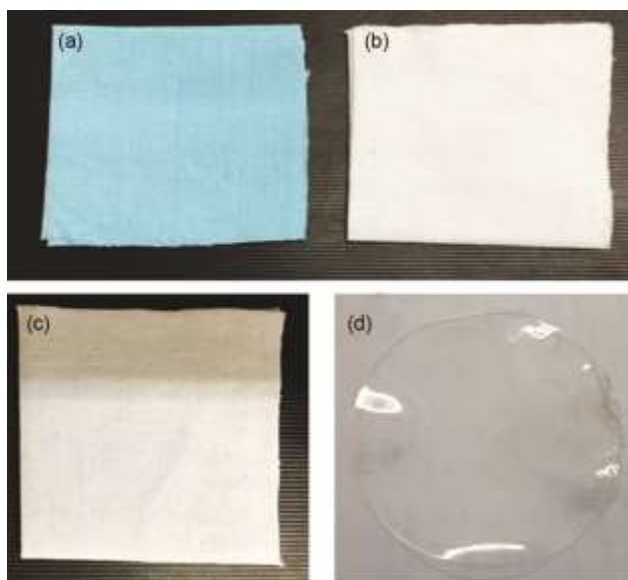


Fig. 3 — Colour deposition on fabric (a) control sample, (b) with PVA-PNO composite film, (c) with directly added PNO and (d) developed PVA-PNO composite film

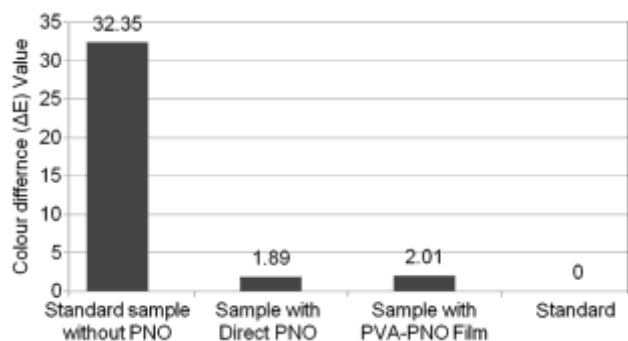


Fig. 4 — Colour difference value of PNO film against reactive blue standard

white sample. The results are provided in Fig 4. The lesser the colour difference value, the better is the performance, and the fabrics are closer to the standard white. A ΔE value of 32.35 is noted for the samples used without DTI polymer and obviously, the sample is found coloured as seen in Fig. 3(a). In the case of white samples with DTI, there is no considerable difference between the direct DTI added to the bath and the developed composite film added to the bath. The colour difference value for the composite film added sample is 2.01 [Fig.3(b)] and the other sample with the direct addition of DTI polymer is 1.89. The results are analysed statistically and no significant difference is found between the samples ($p > 0.05$). This result is achieved mainly due to the excellent performance of the PNO even in the presence of the surfactant/detergent in the wash

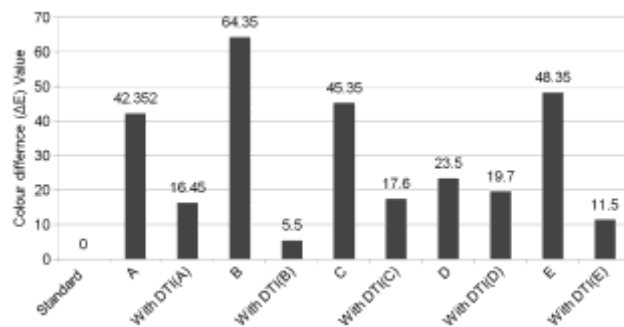


Fig. 5 — Performance of PNO polymer against different dyes [A-acid dyed sample, B-basic dyed sample, C-disperse dyed sample, D-direct dyed sample, and E-addition of all dyes]

liquor. The aromatic structure of the PNO prevents the interaction with the common anionic surfactants present in the detergents unlike other DTI polymers¹³.

3.4.3 Performance of PVA-PNO Film against Different Dyes

As the dye transfer inhibition property of the PNO is noted to be superior in the case of reactive dyes, the performance of PVA-PNO composite film is also evaluated against various commercial dyes, such as direct, acid, disperse, and basic dyes to simulate the real-time washing situation (Fig. 5). It can be observed that the PVA-PNO composite film performs well against all dyes. It is observed that the performance of the film is excellent against basic dyes; and with the acid, disperse, and direct dyes, the performance of the film is noted as good. This difference in performance is due to the chemical nature of the DTI polymer and its interaction with any particular dye. DTI polymer interacts with the dye molecules before it starts reacting with the fibre either by suspending or by solubilising the dye in such a manner that it would not be available for the re-deposition on the fabric². In the case of PNO, the polymer reacts structurally with the dye, solubilises the dye in wash liquor, and prevents any re-deposition. These results are also in line with our previous findings⁴⁴. The general mechanism of interaction between the PNO and the dye is due to the dipole nature of the polymers. Electron density (isosurface studies) reports that the greater delocalization of positive electrostatic potential of aromatic DTI polymers (PNO) in comparison with the aliphatic DTI polymers is the main reason for higher dye inhibition activity. The positive electrostatic potential around H atoms of the aromatic ring and the higher potential on the isosurface (3.1 times the order of magnitude of aliphatic polymer) is reported as a major reason for its superior dye inhibition properties⁴⁹.

Table 2 — Dissolving time of PVA-PNO composite film with and without detergent

Sl. No	Dissolving time, min	
	In the presence of detergent (pH-10)	In the absence of detergent (pH-7)
1	8.5	6.5
2	8.3	7.2
3	9.0	6.8
4	8.5	7.3
5	8.5	7.2
Average	8.56	7

In this case, the performance of the PNO has been evaluated against different dyes (Fig 5). However, the detailed interaction mechanism between the individual dyes and the PNO is not analysed. Carrion⁵⁰ analysed the effect of two different DTI polymers (PVNO and PVP) on the inhibition ability of the direct dyes separately and also by combining them in the presence of different water hardness. They found that a mixture of PVNO and PVP performs better against dye staining in soft water with C.I. Direct blue 200, direct red 243 and direct yellow 86 dyes. Further, in this research, an evaluation is also conducted with the developed film against the mixture of dyes to simulate the real-time condition; the performance of the composite film is found good with a colour difference value of 11.5 (Fig 5). These findings suggest that the performance of the developed PVA-PNO composite film is comparable with direct DTI polymer loading in the wash.

3.5 Analysis of Dissolving Time

The dissolving time of the PVA-PNO composite film is an important factor concerning the proposed application. The average laundry time in a commercial washing machine ranges 30-45 min for a normal one-hour cycle for medium-stained clothes. Hence, it is expected that the developed film should dissolve within the first 10 min completely. The experimental results are provided in Table 2.

The average dissolving time of the PVA-PNO film is found 8.56 min in the wash liquor. The major factor that influences the dissolvability of PVA film is the molecular weight of the film. Previous works²⁸ analysed the factors influencing the dissolving ability of the PVA, where the molecular weight of the polymer plays a vital role in dissolvability. They mentioned that the water-soluble film with desired tensile strength could be produced with an average molecular weight of 50000. In this study, the

molecular weight of the PVA is 85000. This could be the major reason for a higher dissolving time around of 7-10 min. As the role of plasticiser and cross-linking agent are already analysed, further reduction in dissolving time is based only on the chemical nature of the PVA used. The result also shows that the dissolving time of the film increases in the presence of the detergent (Table 2). This is due to the presence of surfactants present in the detergent. These results are found in agreement with the studies of Smith *et al.*³⁰ who reported that the dissolution time of the PVA is greatly affected by the type of surfactant used in the detergents. They mentioned that the presence of cationic surfactant with hydrophobic groups in the wash bath would increase dissolution time significantly. The use of detergent might be the potential reason for the increased dissolution time of the PVA-PNO composite film in the current research. The researcher also mentioned that the rise in wash temperature has reduced the solubility time considerably³⁰. However, in this study, the effect of temperature is not considered. The dissolvability time is calculated at the state of complete dissolution of the PVA-PNO composite film.

4 Conclusion

A dissolvable polymeric composite film has been developed for laundry to dispense dye transfer inhibitors into the wash liquid during the laundry process. The aim of the product is to dissolve the film completely and to release the DTI polymer into the laundry to avoid dye staining, and the dissolving time of the developed film is considered a major criterion in film characteristics. The complete solubility of the film ensures the complete release of the DTI polymer into the laundry liquor and it is essential to arrest the free-flowing dyes in the laundry. The experimental results reveal that the addition of cross-linking agent increases the dissolving time of the film and the plasticiser reduces the same, while the effect of DTI polymer on the dissolving time of the composite film is less. The performance of the developed composite film in terms of resisting the disposition of free-flowing dye on fabric from wash liquor is evaluated and found satisfactory against different dyes, as well as for the mixture of the dyes. The average dissolving time of the developed film is 8.56 min. To reduce the dissolving time further, it is essential to optimise the tensile strength and solubility within the identified narrow range (0-2 mL) of the cross-linking agent

percentage. However, this study provides a potential solution to the problems addressed in the previous research works. At the same time, the effect of the wash temperature and different wash loads on the dissolving ability of the film is not analysed in the current research.

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