



EVALUATION OF PHYSICAL AND CHEMICAL PROPERTIES OF FAS–KPS INDUCED GRAFT CO–POLYMERIZATION OF BINARY VINYL MONOMER MIXTURES ONTO MERCERIZED FLAX

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ABSTARCT

In this work, graft co–polymerization of different vinyl monomer mixtures onto mercerized Flax fibre in presence of ferrous ammonium sulphate – potassium persulphate (FAS–KPS) redox system and evaluation of various parameters like swelling behaviour in different solvents, moisture absorbance and chemical resistance of the grafted and ungrafted fibres has been reported. In the interest of synthesizing bio–degradable and eco–friendly polymers, studies were carried out with different vinyl monomer mixtures; methylmethacrylate (MMA) / Acrylic acid (AA) and MMA / Acrylamide (AAM). Initially optimization of graft yield and graft efficiency was carried out by the variation of different parameters such as reaction time and temperature, concentrations of principal monomer and initiator. Maximum graft yield found with MMA was 138.35% (MMA: 2.94×10^{-3} mol L⁻¹ reaction time: 150 min., temperature: 45°C, FAS–KPS ratio = 0.25:1) Thereafter, the optimization of the monomer concentration of the other component of the binary mixture with MMA as principal monomer was calculated for maximum graft yield (MMA/AA: maximum grafting, 103.55%, AA, 2.19×10^3 mol L⁻¹ and MMA/AAM: maximum grafting, 132%; AAM, 1.76×10^3 mol L⁻¹). Mercerized fibre gave higher graft yield as compared to un–mercerized fibre. The different graft co–polymers, thus prepared, were evaluated for different physical and chemical parameters. Characterization of graft co–polymers was done by making use of spectroscopic techniques and scanning electron microscopy.

Key words: Flax, Methylmethacrylate, Graft copolymer, Swelling, Thermal stability.

INTRODUCTION

Grafting provides a convenient and clean mean for modifying the properties of numerous polymer back–bone and desired properties without drastically affecting the basic properties of the substrate, can be incorporated. The formation of graft copolymer with sufficiently long polymeric sequences of diverse chemical composition opens the way to afford specially

polymeric materials¹⁻⁵. Impregnation of organic and inorganic compounds containing N, P or halogens results in the improvement of thermal stability. Various workers have reported the thermal degradation behaviour and other properties of graft co-polymers⁶⁻⁸.

Since mercerization gives better shining to the polymer surface and results in softening and swelling of the fibre so it was thought important to mercerize the Flax fibre and then undertake graft co-polymerization in different binary vinyl monomer mixtures vis-à-vis un-mercerized fibre. The graft co-polymers thus obtained were subjected for the study of various parameters.

EXPERIMENTAL

Purification of materials

Purification of fibre was done by refluxing it with acetone for 72 h in Soxhlet apparatus⁹. Methylmethacrylate was purified by the method reported in the literature¹⁰. Acrylamide, acrylic acid and potassium persulphate were used as received. Ferrous ammonium sulphate was recrystallized from hot water.

Mercerization of Flax fibre

Flax fibre was immersed in 10% NaOH solution for 2-3 minutes at 35°C and 1% H₂SO₄ was added to it. The fibre was immediately taken out and was given washings with distilled water for removal of acid. However, last washing was given with slightly alkaline softener to neutralize the last traces of acid. The fibre was kept in an air-drier for 15 minutes.

Graft co-polymerization

0.3 g of mercerized Flax fibre was kept immersed in 100 mL of distilled water for 24 h prior to its grafting in air. Known amounts of binary monomer mixture such as MMA/AA and MMA/AAM having MMA as principal monomer (for which optimum reaction conditions were initially worked out) were added to the mercerized fibre. The reaction mixture was stirred constantly for 150 min at 45°C in the presence of optimum concentrations of FAS-KPS as initiator. Separation of homo-polymer / copolymer was done by extraction with suitable solvent: poly (MMA / AA)-g-mercerized Flax with acetone followed by hot water. Graft co-polymer thus obtained was dried at 50°C to a constant weight. The results are reported in Table 1. The % grafting (P_g) and % efficiency (P_e) were calculated as follow:

$$P_g = \frac{W_g - W}{W} \times 100 \quad \dots(1)$$

$$P_e = \frac{W_g - W}{W_m} \times 100 \quad \dots(2)$$

Where W, W_g and W_m are the weights of un-grafted fibre, grafted fibre and monomer.

Swelling, Moisture absorbance, Chemical resistance, Wettability, Dyeing and IR studies

Swelling behaviour of the grafted and ungrafted samples was determined by treating with DMF, H₂O, methanol and n-butanol as per the method reported elsewhere.

Moisture absorbance studies at various humidity levels were carried out as per the method reported earlier¹⁰.

Small wicks of the grafted and ungrafted flax fibres of diameter 5 mm were prepared and initial mark with ink pen was put on the thread and then these were dipped into a beaker containing water. Rise of water through capillary action in each fibre wick was noted down at different time intervals and the percent water up-take (P_w) was calculated as follows:

$$\frac{T_{lb} - D_{tw}}{T_{lb}} \times 100 \quad \dots(3)$$

where T_{lb} = total length of the fibre and D_{tw} = distance travelled by water

100 mg of grafted and ungrafted fibres were put in 100 mL of 1N NaOH and 1N HCl and losses in weights were studied at different time intervals and the percent chemical resistance (P_{cr}) was calculated as:

$$P_{cr} = \frac{T_w - W_{aci}}{T_w} \times 100 \quad \dots(4)$$

where T_w = total weight and W_{aci} = weight after certain interval

0.1% gentian violet solution was prepared in distilled water and 10% common salt and a few drops of acetic acid were added to this solution. The fibre was immersed and the fabric to liquid ratio maintained at 1:50. The samples were kept at room temperature (35°C) for dye absorption. The optical densities of the solutions were determined by digital Photo Colorometer (Model 313 E) at different time intervals of 1, 2, 3, 4 and 5 hrs. The concentration of each solution after dye uptake by the fibre and transmittance (T) were calculated as follows:

$$\text{Transmittance} = \frac{I_i}{I_o} \times 100 \quad \dots(5)$$

$$\text{Concentration of the test solution (after dye uptake)} = \frac{I_i}{I_o} \times \text{Concentration of standard solution} \quad \dots(6)$$

where I_i = optical density of test solution and I_o = optical density of standard solution.

The IR spectra of mercerized flax fibre and its graft co-polymers were recorded on Bomem, Hartmann and Braun (MB-Series) spectrometer.

Evidence of grafting

IR spectra of mercerized flax fibre showed a broad peak at 3420 cm⁻¹ due to bonded -OH and 2925.5, 1458.6 and 1055.87 cm⁻¹ arising from -CH₂, C-C and C-O stretching,