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FABRICATION AND THERMAL PROPERTIES OF PVC

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ABSTRACT

Poly(vinyl chloride) (PVC) films doped with different concentrations of $FeSO_4$ were prepared by solution casting technique and their thermal, structural, morphological, optical and magnetic properties were investigated. Thermal properties of these films were investigated employing differential scanning calorimetry (DSC) and thermo gravimetric analysis (TGA). Using the DSC thermograms, glass transition temperature (T_g) and melting temperature (T_m) of the films were measured. Thermogravimetric curves of the pure and Fe^{3+} doped PVC polymer films showed three distinct steps of weight loss.

INTRODUCTION

Polyvinyl chloride (PVC) is one of the most widely used thermoplastic polymers, with approximately 70% of its global production dedicated to the construction industry. Its durability, chemical resistance, and cost-effectiveness make it an essential material for infrastructure and industrial applications, including water and waste management systems, chemical processing equipment, structural reinforcements, and insulation materials. Beyond construction, PVC plays a critical role in medical and consumer products, such as blood bags, medical tubing, credit cards, and packaging materials, owing to its versatility and adaptability. PVC is further recognized for its high chemical and thermal resistance, low cost, barrier properties, and ease of production, making it a preferred material for applications ranging from sensors to electrical equipment design [1, 2].

DSC (Differential Scanning Calorimetry) and TGA (Thermogravimetric Analysis) are key techniques used in scientific journals to study Polyvinyl Chloride (PVC) and its blends/composites, analyzing its thermal stability, glass transition (), melting/decomposition points, and interactions with additives (like fillers, plasticizers, etc.), revealing crucial information for optoelectronic, stabilization, and failure analysis applications DSC measures the temperature and heat flow associated with transitions in materials as a function of temperature or time in a controlled atmosphere. Differential scanning calorimetry (DSC) is an effective analytical tool for characterizing the physical properties of a polymer. DSC enables determination of melting, crystallization, and mesomorphic transition temperatures, and the corresponding enthalpy and entropy changes, and characterization of the glass transition and other effects which show either changes in heat capacity or a latent heat[3]. Calorimetry takes a special place among other methods. Poly(vinyl chloride) (PVC) is a polymer with low thermal stability and would undergo degradation under elevated processing temperature [4].

This study presents, for the first time, a comprehensive investigation of the synthesis, and thermal behavior of PVC+$FeSo_4$ polymer. The findings demonstrate that PVC+Fe^{3+} possess intriguing properties that can expand the application range of polymers. This research provides valuable insights into the interplay between Ferrous and PVC matrices and opens new pathways for their utilization in advanced industrial applications.

MATERIALS AND METHODS

Poly(vinyl chloride) (PVC) obtained from M/S Sigma Aldrich has a mean relative molecular mass of about 534,000 g/mol. PVC polymer films doped with $FeSO_4$ in various concentrations were prepared at room temperature by solution casting method. The desired concentration of $FeSo_4$ solutions (1, 2, 3, 4 and 5%) were prepared by using distilled water. 1g/mol of PVC polymer is dissolved in tetra hydro furan (THF) separately. Different amounts of $FeSo_4$ solution (1, 2, 3, 4 and 5 mol%) were added into the polymer solution. The mixture was magnetically stirred for 10-12 hours to get homogeneous mixture and then cast onto plastic dishes. The film was slowly

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evaporated at room temperature to obtain free standing polymer film at the bottom of the dishes.

In order to investigate the nature of the prepared polymer films, differential scanning calorimetry (DSC) measurement was carried out by a SEIKO calorimeter (DSC–220) with continuous heat rate of 10 °C/min under nitrogen atmosphere from 50 to 400 °C. The thermo gravimetric analysis (TGA) was done using the SEIKO thermal analysis (TGA–20) system in the presence of nitrogen flow from 50 to 500 °C, at the heating rate of 10 °C/min.

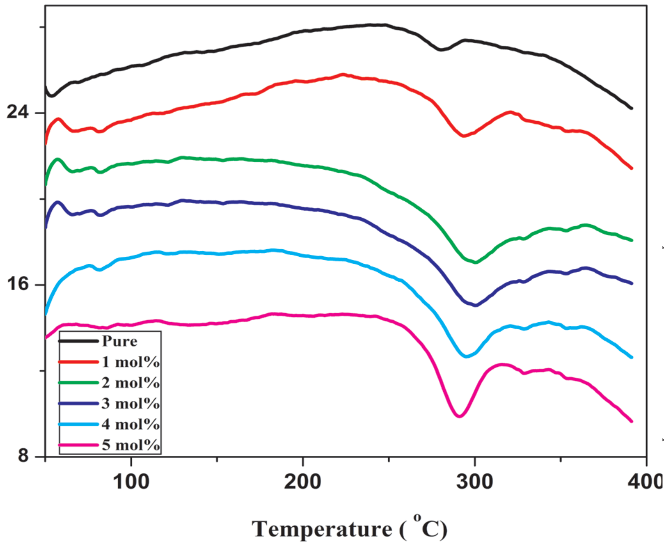

Figure. 1. DSC curves of pure and Fe^{3+} doped PVC polymer films

From Figure 1, it can be seen that all the samples exhibit only one T_g which indicates the homogeneous behavior of polymer film. Another endothermic peak has been observed at 280 °C and 290 °C for pure, and Fe^{3+} doped PVC films respectively, which is due to dehydrochlorination of PVC. In pure PVC, beyond T_g, the plot remains virtually invariant until the initial stage of another endothermic peak observed around 280 °C. This is the melting point of pure PVC [7, 8]. The melting temperature T_m of Fe^{3+} doped PVC electrolyte was found to vary from 289 to 293 °C, with higher T_m corresponding to lower Fe^{3+}content. This positive deviation in T_m for doped PVC indicates a strong interaction between the doped Fe^{3+} and PVC films which increases the spherulite size and their surface free energy [9]. In addition, the area under the melting peak increases as the Fe^{3+} content increases. This clearly indicates that the micro movement of the PVC chain becomes easier by the addition of the Fe^{3+}, presumably owing to the decrease in crystallinity of the electrolytes [10]. A slight decrease of T_m towards lower temperatures for 4 and 5 mol% of Fe^{3+} reveals the disruption of crystallinity of the host polymer.

TGA analysis

Thermal stability of the polymer composites plays an important role in determining the limit of their working temperature and the environmental conditions for use, which are related to their thermal decomposition temperature and decomposition rate [11]. TGA helps in providing a method for thermal stability testing and provides complementary and supplementary information to that obtained from the most commonly used thermal technique, DSC.

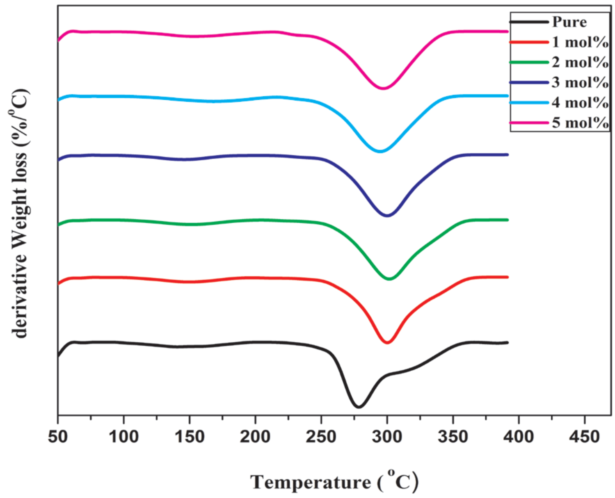

Figure. 2. DTG curves of pure and Fe^{3+} doped PVC polymer films

Figure. 2 shows the DTG curves of pure and Fe^{3+} (1-5 mol%) doped PVC films. Thermal stability is represented by the weight loss of the sample after heating over the temperature range 40 – 400 °C. Analysis of TGA curves reveals that there are three distinct steps of degradation. A first degradation is observed from 50 to 70 °C with a weight loss of 3% which may be due to the evaporation of THF and residual moisture absorbed. The second degradation in the temperature range 70 – 250 °C is due to the residual solvent evaporation and the glass transition of the polymer electrolyte sample, where the percentage weight loss for the system is 18%. The third stage of weight loss in the temperature range 250 – 400 °C is attributed to dehydrochlorination [12] where the molecular reaction and radical propagation reactions become more probable and contribute to the cleavage of C – Cl bond. At the temperature higher than 310 °C, the chain radical mechanism becomes relevant and the melting of C –C or C – H bands takes place. The degradation temperature increases with increasing dopant concentration. Probably, the presence of ferrous strengthens the van der waals interactions between the PVC chains, which leads to increased decomposition temperature.

The first and second decomposition temperature (T_{onset} I, T_{onset} II), and the Char yield (residual material) are summarized in Table 1. Results show that degradation temperatures (at first degradation) of Fe^{3+} doped films have increased compared to pure PVC. This increase can be explained with respect to the catalytic property of Fe^{3+}[13] which accelerate the dehydrochlorination of PVC chains. In addition, as shown in Fig. 2 the residuals of polymer films are greater than that for pure PVC. This indicates that utilizing Fe^{3+} into PVC enhances the char yield/carbonization of the polymer. The char yield is the standard method for evaluation of limiting oxygen index (LOI) of a polymer in accordance with Van Krevelen and Hoftyzer equation [14].

$$LOI=17.5+0.4\ Yc \quad \ldots\ldots\ldots\ldots(1)$$

where Yc is char yield, and LOI is the minimum oxygen concentration. A polymer with high LOI has low flammability. LOI for the PVC films containing Fe^{3+} (1-5 mol%) is presented in Table 1. LOI values are calculated to 26, 29, 29, 30, 30 and

31 respectively. From the above discussion, it is concluded that the thermal stability limit of the pure and Fe^{3+} doped PVC film is about 340 °C [15].

Table 1. Thermogravimetric results for pure and Fe^{3+} doped PVC films

PVC:Fe^{3+} concentration (mol %)	T_{onsetI} (°C)	$T_{onsetII}$ (°C)	Charyield(%) at 400 °C	LOI
Pure	109	235	21	25.9
1	113	247	28	28.7
2	114	253	29	28.7
3	120	254	29	29.9
4	124	242	31	30.3
5	122	254	32.5	30.5

CONCLUSION

Poly(vinyl chloride) (PVC) films filled with different concentrations of FeSO 4 were prepared by solution casting technique. Thermal properties of these films were investigated. Thermal properties of these films were investigated with differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Using the DSC thermograms, glass transition temperature (T g) and melting temperature (T m) of PVC films were measured. Thermogravimetric curves of the pure and Fe 3+ doped PVC polymer films showed three distinct steps of weight loss. Thermogravimetric studies also show that the thermal stability of the pure and Fe 3+ doped PVC films occurs at 340 o C.

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