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Comparative study of Flame retardant and thermal properties of PVC/MMT and PVC/LDH nanocomposites

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ABSTRACT: Nanocomposites of poly (vinyl chloride) (PVC) were prepared to increase the flame retardant property. The solution cast method was used for the formulation of PVC nanocomposites, using nano fillers such as montmorillonite (MMT) and layered double hydroxide (LDH). The resulting nanocomposites were analyzed by means of thermogravimetry analysis (TGA), static oven test and flame retardant properties. The obtained results were compared with each other and also with pristine PVC film. The results of thermal analysis, static oven test and flame retardant properties indicated that the nano sized MMT as well as LDH work as flame retardant for the PVC formulations.

KEYWORDS: Poly (vinyl chloride), Montmorillonite, Layered double hydroxide, Nano-composite, Thermogravimetry analysis, Flame retardant.

I.INTRODUCTION

Nanocomposites are materials having unique physical properties, mechanical properties and wide application potential in various field. These nanocomposites materials pusesing the different some physical and chemical properties as compared to both the pure polymers and nanoparticles.Synthesised nanocomposites are obtained by encapsulating nanoparticles inside the polymer[1]. Poly (vinyl chloride), PVC, has industrial importance and used in large scale worldwide due to its outstanding properties and versatility. Due to its unique structure and reactivity together with high strength, stiffness, swelling behavior, and large surface area widely used in different fields such as agriculture, medical etc. PVC can be plasticized by different plasticizers with different amount, so that it can be made from hard, rigid as well as soft. Being thermoplastic, PVC is used in an extensive variety of applications such as packaging, sheets, pipes, cable insulation, molded articles, flooring and medical equipment.

In general, there are three main methods to prepare polymer/ clay nanocomposites: in situ polymerization, solution blending and melt blending. The main goal is to disperse the clay layers into the polymer matrix in order to obtain exfoliated structures. Several studies using the in situ polymerization, solution blending, and melt blending processes, using either the natural clay or organically modified clay, have appeared since Ishida and co-workers reported a novel approach to nanocomposite preparation in which PVC was blended with a tin stabilizer in the presence of clay. However, only few successful works have been carried out on PVC/montmorillonite nanocomposites, and practically all of these are limited to plasticized PVC.[2]

In present work we were enhancing the flame retardant and thermal properties of PVC material by using nano filler like MMT and LDH. The comparative study of these nanocomposites according to flame retardant and thermal properties of PVC was done.



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II.EXPERIMENTAL WORK

Material:

A suspension grade Polyvinyl Chloride (PVC) Reon grade: K 67, Reliance Industries limited, India was used as received. Commercial grade layered double hydroxides (LDH) (Mg-AL based) as Hydrotalcite (HYDT) and Montmorillonite (MMT) (Al-Si) based were used as PVC synergists obtained from Sigma Aldrich as received without any further modification. Cyclohexanone (97%) was procured from Loba chemicals and used without any further purification.

Procedure

Dissolution of PVC

5 g of PVC (K-67) powder was added slowly pinch by pinch in to 250 ml beaker containing 10 ml of cyclohexane. Further 30 ml cyclohexane was added and the solution was heated slowly at 400C for complete dissolution of PVC. During this process PVC powder swells and then got dissolved in solvent.

Dissolution of Nanoparticles

Nanoparticle of LDH with varying 0, 2, 4, 6, 8 and 10 phr was added respectively in 5 ml of cyclohexane in beakers. The solution was sonicated for 10 minutes at 40OC for complete dissolution of LDH/MMT nanoparticles. Same dissolution procedure was used for MMT nanoparticle. The formulation was mentioned in table No.1 Table No.1: Formulation of PVC and Nanoparticle.

Sr.No.	Label No.	PVC(g)	LDH or MMT (phr)	Cyclohexanone (ml)
1	L-1	5	0	35
2	L-2	5	2	35
3	L-3	5	4	35
4	L-4	5	6	35
5	L-5	5	8	35
6	L-6	5	10	35

Preparation of Nanocomposite film

The stock solutions were made from PVC and LDH nanoparticles separately.LDH/MMT nanoparticles solution were added in to PVC solution. The mixture of LDH and PVC solution stirred vigorously with glass rod to assure homogeneity. Homogeneous solution then poured in to petry plate and solvent allowed to evaporate at room temperature for 15 hours. Same dissolution procedure was used for MMT nanoparticle.

Characterizations:

Thermal stability of PVC by Thermo gravimetric Analysis (TGA)

Thermal degradation behavior of PVC samples was analyzed with PerkinElmer 4000 thermo gravimetric analyzer. All experiments were performed within the temp armature range of 100 0C. The heating rate of the samples was kept constant at 15 0C/minute under nitrogen gas environment. It has been reported that dehydrochlorination phase (loss of HCl) of PVC could be easily studied by TGA, as it is the first and most promonent stage of mass loss during PVC degradation. The change in onset temperature was considered as a criterion for improvement in the thermal stability of



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PVC. TGA were carried out to estimate and investigate the thermal stability of PVC composite and the influence of filler (LDH, MMT) on thermal stability of PVC composite.

Hardness Test

Hardness is defined as the resistance of a body to penetration caused by another body. We had hard material and we had to use Shore D for hardness testing. Sample of film of PVC/LDH having 3 cm length and 1 cm breadth is taken. This sample was placed on Shore D hardness tester such that indenture position was approximately at centre of the specimen and the indenture just touches the sample. The button was pressed which lift the base plate and the reading observed.

Flame Test

It is the distance travelled by the flame over a sample in one minute. In this the sample is held horizontally with its transfer axis inclined at an angle of 450 to the horizontal then flame is applied for specific duration to the free end of the test specimen and removed. The time taken by flame to travel a distance of 100 mm is recorded. The time required to burn 10 mm length of the sample is proportional to its burning characteristics. The sample of PVC/LDH of length 3 cm, width 1 cm and thickness 1.2 cm was taken; a line was marked of a distance of 2 cm from both sides. The sample was hold in such a way that it should be horizontal and about 6 mm above the wire gauze. The burner was pulled to extreme right position and adjusts the flame height about 10 mm. The observation is taken whether It catches fire or extinguishes before reaching mark of the sample and the time was recorded when the fire extinguished.

III.RESULTS AND DISCUSSION

1. Hardness Test

The readings given in table shows that the hardness of PVC/LDH is greater than PVC/MMT. The films of PVC/LDH nanocomposite are harder than PVC/MMT nanocomposite. The hardness increased with increased in phr of filler in composite of PVC.

Table: 3					
Sr.No.	Filler(phr)	Sample-1	Sample-2		
		PVC/LDH	PVC/MMT		
1	0	33.8	33.8		
2	2	34.7	34.0		
3	4	35.1	35.0		
4	6	35.6	35.5		
5	8	36.6	36.2		
6	10	46.6	43.6		



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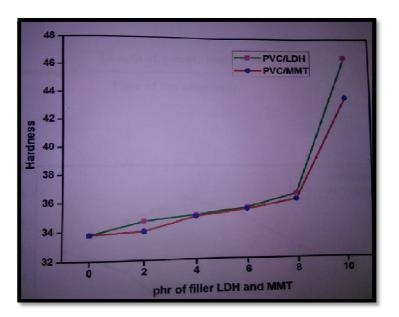
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2. Flame Test:

The following table-4 shows that the rate of burning of PVC/LDH is less than rate of burning of PVC/MMT. This states that PVC/LDH nanocomposite has high flame retardancy than PVC/MMT.Burning if LDH produces a mixture of metal oxides and carbineous char that acts as flame retardant. LDH shows self extinguishing behavior. Dimentions of sample is $30\text{mm} \times 10\text{mm} \times 1.2\text{mm}$. While the rate of burning is determined by using the following formula.

The rate of burning= $\frac{\text{length of smaple (mm)} \times 60}{\text{Time of burning (s)}}$

Table:4						
Sr.No.	Filler(phr)	Sample-1 PVC/LDH Rate of burning (mm/s)	Sample-2 PVC/MMT Rate of burning (mm/s)			
1	0	24.0	24.0			
1	0	24.0	24.0			
2	2	22.5	26.0			
3	4	18.9	27.7			
4	6	16.3	26.47			
5	8	15.0	24.0			
6	10	13.3	22.7			



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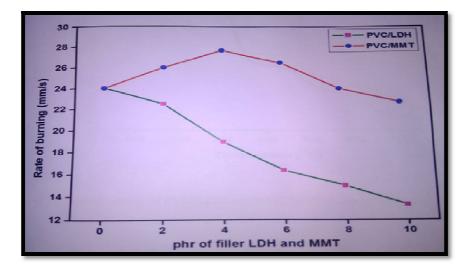
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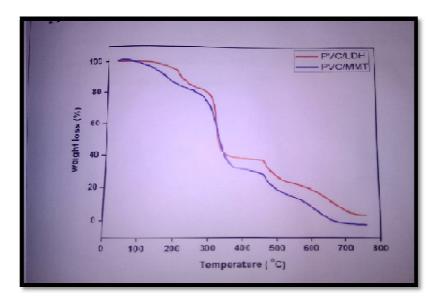


3. Thermal Gravimetric Analysis

All the films were degraded in three steps, the first step degradation was stared 1500c to 2470C corresponding ti weight loss 13% that may due to dehydrochlorination of PVC and dehydration of LDH. The second step degradation observed in the range of 2400C to4230C with of 47% weight loss. Second step is smooth and resulting from dehydrocarbonation and oxidation of PVC. The third step thermal degradation reported in the thermal range 4240C to7630C and weight loss in the range of 34% observed due to the dehydrolation and decarbonation of LDH.

PVC/MMT films were degraded in three steps, the first degradation was started 1500C to 2250C corresponding ti weight loss 9.38% that may be due to evolution of interspaced water molecules. The second step degradation observed in the range of 2250C to4380C with of 54.79% weight loss due to evolution of organic substances. The third step thermal degradation reported in the temperature range 4370C to 7630C and weight loss in the range of 31.16% observed due to the dehydroxylation.

TGA Garph





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IV.CONCLUSION

The PVC nanocomposits were prepared by solution blending method the prepare nanocomposites from LDH shows better flame retardance properties than MMT. The hardness of LDH loaded nanocomposites were found quite than MMT loaded nanocomposites both type of nanocomposites showed three stage degradadtion. The study revealed the addition of LDH with PVC gives further enhancement of stability, time as well as retardation of discoloration of PVC strip as compare to MMT and PVC.

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