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Development of Optimum Combination of MMT Clay Reinforced Composite

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Abstract*: The Montmorillonite (MMT) clay toughened epoxy (85%) / unsaturated polyester (15%) polymer blend system is developed. Studies on Mechanical, Thermal and Damping properties are conducted to determine the effect of the variations of MMT clay on the polymer blend. The Mechanical property studies showed that 3% clay reinforced polymer blend system exhibited optimum results, while the Damping property studies showed that 4% clay reinforced polymer blend system exhibited optimum results and Thermal property studies revealed that 5% clay reinforced polymer blend system exhibited optimum results. The prime objective of this work is to validate the obtained experiment results and to identify the best-fit combination. The results are validated by using Design Expert Version 8 Software. Validation of the experimental results, indicate negligible variations with the software generated results. Since different variations of clay percentages are achieved for different properties, a decision table is constructed to determine the best-fit combination suitable for the selected applications.

***Keywords*: Mechanical, Thermal, Damping, Blend, Optimum.**

I. INTRODUCTION

A study on the effect of Montmorillonite (MMT) clay on the mechanical, thermal and damping properties of epoxy/unsaturated polyester (UP) (85/15 %w/w) blended nanocomposite is conducted and published by the author in reputed journals [1], [2]. Blending of resins is done to improve the mechanical properties (in this case toughness property). Epoxy, even though it has very good mechanical properties but has poor toughness. So, polyester which has better toughness property is blended with epoxy resin, to improve its toughness property.

A.Varadarajulu [3] have conducted miscibility studies of blends of epoxy with unsaturated polyester resin in chloroform are carried out by viscosity, ultrasonic velocity, and refractive methods at 30°C. By using viscosity data, the interaction parameters are computed, which indicated that epoxy/unsaturated polyester resin blends are miscible. The miscibility is further confirmed by the ultrasonic velocity and refractive index methods. It also confirmed that 85%-15% of epoxy-polyester gave better miscibility results. Studies by A. Benny Cherian [4] has indicated that epoxy resins show good miscibility and compatibility with unsaturated polyester resin on blending and that there is substantial improvement in the toughness and impact resistance. Considerable enhancement of tensile strength and toughness is also indicated in their studies. Their studies also indicated that epoxy/UP resin blends showed substantial improvement in thermal stability as evident from TGA and DSC studies. Kornmann [5] synthesized Montmorillonite (MMT) clay and investigated its effect on unsaturated polyester (UP). Montmorillonite (MMT) is derived from bentonite, purified, activated by sodium ions and mixed with reacting unsaturated polyester (UP). At a MMT content of only 1.5 vol. %, the fracture energy, of the nanocomposite is doubled, 138 J/m^2 as compared with 70 J/m^2 for the pure UP. Chinnakkannu Karikal Chozhan et al [6] developed and studied unsaturated polyester toughened epoxy matrix systems with varied concentration. The thermal properties namely Tg, HDT of unsaturated polyester–epoxy systems have been compared with those of unmodified epoxy systems. The reduction in the values of Tg for unsaturated polyester toughened epoxy system is due to the flexibility imparted by unsaturated polyester to the epoxy matrix. The mechanical studies inferred that the incorporation of unsaturated polyester into epoxy resin enhanced the values of impact strength. Homogeneous microstructures of SEM confirmed the effective interaction of organoclay with UP–epoxy resin.

This paper validates the experimentally determined results for the effect of clay content on the mechanical properties, thermal properties and damping properties of epoxy / unsaturated polyester blend using Design Expert software. Moreover a decision table is constructed to identify the best-fit combination of epoxy/polyester/clay suitable for the selective applications.

II. METHODOLOGY

A. Materials

The resins used in this study are Epoxy (LY-556) and hardener (HY-951) blended with Unsaturated Polyester, with 2% cobalt naphthanate as accelerator, 2% Methyl ethyl ketone peroxide (MEKP) as catalyst in 10% Dimethylaniline (DMA) solution as promoter, in the ratio of the resin/ accelerator/ catalyst/ promoter:100/2/2/2.

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In addition, exfoliated montmorillonite clay (product No.:682608; brand: Aldrich, USA; product name: Nanoclay, 1.28E; formula: $H_2Al_2O_6Si$; Molecular weight: 180.1 g/mol; Appearance (Colour): white; Appearance (form): powder; loss on drying: ≤18.0%; density: 600-1100 kg/m^3; size: ≤ 25 microns) is surface modified with 25-30% trimethyl stearyl ammonium, is used as filler material.

B. Method

Clay is dried in an oven at a temperature of 80 °C for 24 hours. The polymer blends of epoxy and polyester are mixed using a Haake rheometer at 150°C and 50 rpm for 10 min. Then pre-calculated amount of dried clay [7] is mixed with epoxy/UP (i.e. 85/15 %w/w ratio) blend in a suitable beaker. Clay is mixed in stipulated quantity (i.e.. 0%, 1%, 2%, 3%, 4% and 5%) to the epoxy/UP blend and is mixed thoroughly using a mechanical shear stirrer for about 1 hour at ambient temperature conditions. Then the mixture is placed in a high intensity ultra-sonicator for one and half hour with pulse mode (15s on / 15s off). External cooling system is employed by submerging the beaker containing the mixture in an ice bath to avoid temperature rise during the sonication process. Once the process is completed, hardener/accelerator/catalyst/promoter (100:10/2/2/2) parts by weight is added to the modified epoxy/UP/clay mixture. A glass mould with required dimensions is used for making specimens on par with ASTM standards. The glass mould is coated with mould releasing agent to enable easy removal of the specimen. The nanocomposite mixture is poured over the glass mould. Brush and roller are to impregnate the nanocomposite. The closed mould is kept under pressure for 24 hours at room temperature. To ensure complete curing the blended nanocomposite specimens are post- cured at 70°C for 1 hour. The removed specimens are cut into specimens of required size and shape in accordance to ASTM standards for testing. Direct processing technique has been used for the above nanocomposites preparation.

Characterization of the nanocomposite encompasses mechanical (tensile, flexural, hardness and impact tests), thermal (TGA, DSC tests) and vibration analysis. The obtained results are validated by using Design Expert Version 8 Software. Validation of the experimental results, indicate negligible variations with the software generated results. Since different variations of clay percentages are achieved for different properties, a decision table is constructed to determine the best-fit combination suitable for selective applications.

III. RESULTS AND DISCUSSIONS

A. Validation of mechanical properties

Table I shows the obtained experimental data for the effects of clay content (process variable under study) on the mechanical properties of epoxy/ unsaturated polyester/ MMT clay nanocomposites.

Table-I: Experiment data considered for validation

CC %	TS MPa	IS J/m	FS MPa	H Rc
0	20.71	4.61	5.12	105
1	24.31	4.92	6.69	108
2	26.29	5.15	7.34	115
3*	**29.71**	**5.49**	**10.67**	**120**
4	27.26	5.23	9.35	108
5	25.28	5.04	8.63	95

*- optimum value, CC- Clay Content, TS – Tensile Strength, IS – Impact Strength, FS – Flexural Strength, H – Rockwell hardness.

Tensile strength

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over tensile strength are presented in Figure 1. From these results, a

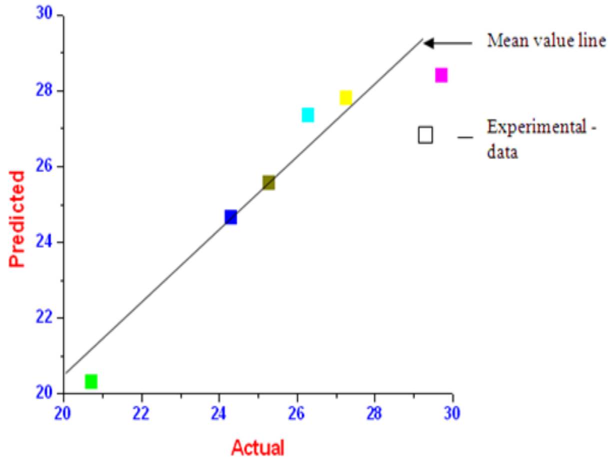

Fig.1. Predicted (software generated) Vs actual (experimental) values of tensile strength

Table-II: Predicted Vs Actual values of tensile strength

Run	CC %	TS (actual values) MPa	TS (predicted values) MPa
1	0	20.71	20.31
2	1	24.31	24.66
3	2	26.29	27.36
4	**3**	**29.71**	**28.41**
5	4	27.26	27.81
6	5	25.28	25.56

suitable model is selected. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on the tensile strength of epoxy/ unsaturated polyester/MMT clay nanocomposites. The quadratic function obtained is given in Equation (1). This equation is used to generate the predicted values by the software as shown in Table II.

$$TS = 20.31028 + 5.173019 * CC - 0.82448 * CC^2 \quad (1)$$

Impact strength

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over impact strength are presented in Figure 2. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on epoxy/ unsaturated polyester / MMT clay nanocomposites. The quadratic function obtained is given in Equation (2). This equation is used to generate the predicted values by the software as shown in Table III.

$$IS = 4.556922 + 0.506789 * CC - 0.08075 * CC^2 \quad (2)$$

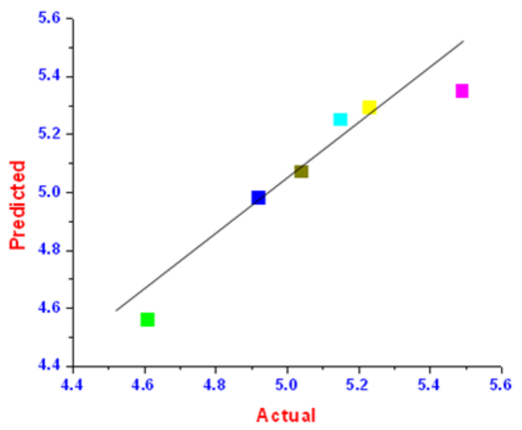

Fig.2. Predicted Vs actual values of impact strength

Table-III: Predicted Vs Actual values of impact strength

Run	CC %	IS (actual values) J/m	IS (predicted values) J/m
1	0	4.61	4.56
2	1	4.92	4.98
3	2	5.15	5.25
4	**3**	**5.49**	**5.35**
5	4	5.23	5.29
6	5	5.04	5.07

Flexural strength

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over flexural strength are presented in Figure 3. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on epoxy/ unsaturated polyester / MMT clay nanocomposites. The linear function obtained is given in Equation (3). This equation is used to generate the predicted values by the software as shown in Table IV.

FS = 5.915135 + 0.806757 * CC (3)

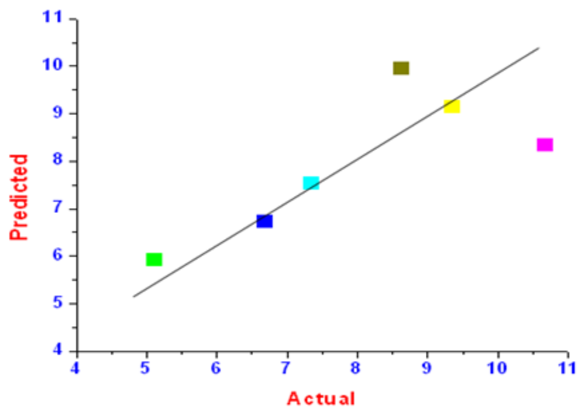

Fig.3. Predicted Vs actual values of flexural strength

Table-IV: Predicted Vs Actual values of flexural strength

Run	CC %	FS (actual values) MPa	FS (predicted values) MPa
1	0	5.12	5.92
2	1	6.69	6.72
3	2	7.34	7.53
4	**3**	**10.67**	**8.34**
5	4	9.35	9.14
6	5	8.63	9.95

Hardness

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over hardness are presented in Figure 4. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on epoxy/ unsaturated polyester / MMT clay nanocomposites. The cubic function obtained is given in Equation (4). This equation is used to generate the predicted values by the software as shown in Table V.

H = 105.027 - 1.57727 * CC + 5.582884 * CC^2 - 1.13365 * CC^3 (4)

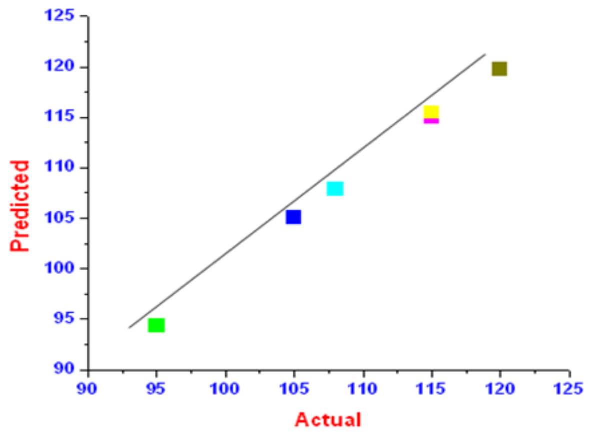

Fig.4. Predicted Vs actual values of hardness

Table-V: Predicted Vs Actual values of hardness

Run	**CC %**	**H (actual values) Rc**	**H (predicted values) Rc**
1	0	105	105.03
2	1	108	107.87
3	2	115	115.03
4	**3**	**120**	**119.71**
5	4	115	115.49
6	5	95	94.38

B. Validation of thermal properties

Table VI shows the obtained experimental data for the effects of clay content (process variable under study on the thermal property of epoxy/ unsaturated polyester / MMT clay nanocomposites.

Table-VI: Experiment data considered for validation

CC %	T °C
0	430
1	432
2	433
3	433
4	431
5*	**429**

*- optimum value, T – Glass transition temperature

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over Glass transition temperature of the nanocomposite are presented in Figure 5. From these results, a suitable model is selected. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on epoxy/ unsaturated polyester / MMT clay nanocomposites. The quadratic function obtained is given in Equation (5). This equation is used to generate the predicted values by the software as shown in Table VII.

$$T = 429.942563 + 2.72238586 * CC - 0.58100147 * CC^2 \quad (5)$$

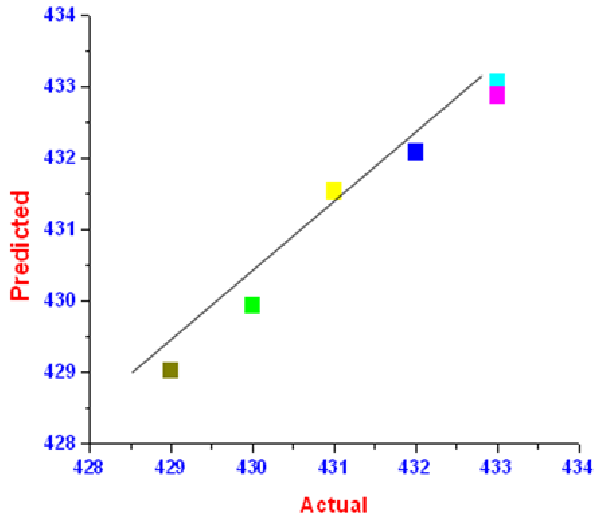

Fig.5. Predicted Vs actual values of glass transition temperature

Table-VII: Predicted Vs Actual values of glass transition temperature

Run	CC %	T (actual values) °C	T (predicted values) °C
1	0	430	429.94
2	1	432	432.08
3	2	433	433.06
4	3	433	432.88
5	4	431	431.54
6	**5**	**429**	**429.03**

C. Validation of damping property

Table VIII shows the obtained experimental data for the effects of clay content (process variable under study) on the damping ratio of epoxy/ unsaturated polyester / MMT clay nanocomposites.

Table-VIII: Experiment data considered for validation

CC %	dr
0 (mode 2)	2.13
0 (mode 3)	1.37
1 (mode 2)	2.47
1 (mode 3)	1.9
2 (mode 2)	1.92
2 (mode 3)	1.37
3 (mode 2)	1.97
3 (mode 3)	1.5
***4 (mode 2)**	**2.62**
***4 (mode 3)**	**2.16**
5 (mode 2)	1.69
5 (mode 3)	1.66

* - optimum value, dr – damping ratio

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over Damping ratio of the nanocomposite presented in Figure 6. From these results, a suitable model is selected. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on epoxy/ unsaturated polyester / MMT clay nanocomposites. The quartic function obtained for mode 2 and mode 3 is given in Equation (6). This equation is used to generate the predicted values by the software as shown in Table IX.

$$dr = 2.13190476 + 1.7593254 * cc - 2.05327381 * cc^2 + 0.70555556 * cc^3 - 0.07375 * cc^4 \quad (6)$$

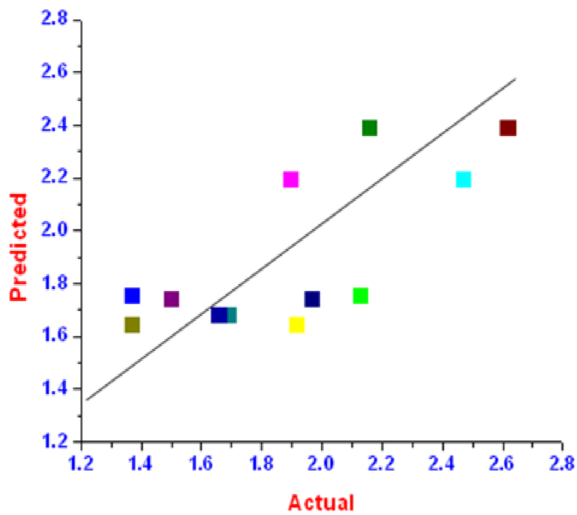

Fig.6. Predicted Vs actual values of damping ratios

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Table-IX: Predicted Vs Actual values of damping ratios

Run	CC %	dr (actual values)	dr (predicted values)
1	0	2.13	1.75
2	0	1.37	1.75
3	1	2.47	2.19
4	1	1.9	2.19
5	2	1.92	1.64
6	2	1.37	1.64
7	3	1.97	1.74
8	3	1.5	1.74
9	**4**	**2.62**	**2.39**
10	**4**	**2.16**	**2.39**
11	5	1.69	1.68
12	5	1.66	1.68

Table X gives the summation of results indicating the actual (experiment) values and the predicted (system response) values of all the properties considered for study. Validation of the experimental, results indicate negligible variations with the software generated results. This proves that the experimental results are correct and can be considered as best-fit values.

Table-X: Summation of results

Property	Objective	Experimental values %	Software predicted values %
Mechanical	Maximize mechanical properties	3	3.4
Thermal	Minimize glass transition temperature	5	5
Damping	Maximize damping ratio	4	4.27

It is observed from Table X, that there is difference in the optimum values of the nanocomposite obtained, for the different properties considered in the study.

- In case of mechanical properties the maximum strength is observed at 3 wt. % clay content.
- In case of thermal properties maximum thermal stability is observed at 5 wt. % clay content.
- In case of damping property, maximum damping ratio is observed at 4 wt. % clay content.

To arrive at an optimum combination for the nanocomposite material, a decision table is considered as the tool. Here the epoxy/UP/clay nanocomposite developed possesses better mechanical and thermal properties and can be applied in areas as given below [8]:

(i) Automobile components and accessories like control panel boards, interior parts of automobile etc..
(ii) Fire retardants and fire resistant cables
(iii) Superior, low-cost film materials.

Weightage (out of 10) is given for the properties based on their importance. For the above applications, mechanical properties like tensile, flexural and impact are given prime importance. Also thermal stability is given more importance. The remaining properties like hardness and damping are given relatively less importance. Based on this, a decision table is constructed as shown in Table XI.

Here values like b_1, b_2, b_3 etc. and $a * b_1$, $a * b_1$, $a * b_2$, $a * b_3$ etc.. are calculated. Sample calculations are given below.

(i) Consider the tensile strength (TS) at 3% clay content (CC). Here comparison is made with the pure blend. Here b_3= TS at 3% CC / TS at 0% CC = 29.71/20.71 = 1.43. Similarly b_1, b_2, b_4 and b_5 are calculated. In the same way calculations are performed for other mechanical properties.

(ii) Consider glass transition temperature (Tg) at 3% CC. Since the objective here is to minimize the Tg, Here b_3= Tg at 0% CC / Tg at 3% CC = 430/433 = 0.99. Similarly b_1, b_2, b_4 and b_5 are calculated are calculated.

TABLE-XI: DECISION TABLE

Properties	Weightage (a)	Priority value (a x b_i) [i =1,2 and 3]									
		1%CC		2%CC		3% CC		4% CC		5% CC	
		(b_1)	($a*b_1$)	(b_2)	($a*b_2$)	(b_3)	($a*b_3$)	(b_4)	($a*b_4$)	(b_5)	($a*b_5$)
Tensile strength	10	1.17	11.7	1.27	12.7	1.43	14.3	1.32	13.2	1.22	12.2
Flexural strength	10	1.31	13.1	1.43	14.3	2.08	20.8	1.83	18.3	1.69	16.9
Impact strength	10	1.07	10.7	1.12	11.2	1.19	11.9	1.13	11.3	1.09	10.9
Hardness	8	1.03	8.24	1.09	8.72	1.14	9.12	1.05	8.4	0.9	7.2
Thermal stability	10	1.25	12.5	1.35	13.5	1.5	15	1.6	16	1.75	17.5

Glass transition temperature	9	1	9	1.01	9.09	0.99	8.91	0.99	8.91	1	9
Damping ratio (mode 2)	8	1.16	9.28	0.9	7.2	0.92	7.39	1.23	9.84	0.79	6.32
Total			82.22		83.71		**93.72**		91.55		85.42

So it is concluded that 85% Epoxy/ 15% unsaturated polyester nanocomposite with 3% clay content is the best-fit material suitable for the above considered applications.

IV. CONCLUSIONS

In the present work, a study on the effect of montmorillonite (MMT) clay on the mechanical, thermal and damping properties of epoxy/unsaturated polyester (UP) (85/15 %w/w) blended nanocomposite is conducted. To study the effect of clay on the blended nanocomposite, the resin blend is mixed with clay in varying proportions, the nanocomposite is studied by varying the clay content as 0% (pure blend), 1%, 2%, 3%, 4% and 5%. The following conclusions are drawn from the study.

1. The mechanical properties of the clay filled epoxy/unsaturated polyester nanocomposite are observed to steadily increase and is found to be maximum at 3 wt. % clay content (CC), when compared to other variations. The reasons for the mechanical strength values to fall at 4wt. % and 5 wt. % clay contents, is due to increase in viscosity in the blended nanocomposite which causes poor dispersion of nanoparticles. Under the present processing conditions, it is observed that increase in viscosity is due to addition of more nanoclay, which makes the resin degassing difficult. This leads to the entrapment of small air voids within the blend, which also causes poor dispersion of the clay, resulting in the formation of agglomerates in the epoxy/UP blend matrix.
2. The thermal properties of the clay filled epoxy/unsaturated polyester nanocomposite are observed to steadily increase and is found to be maximum at 5 wt. % clay content, when compared to other variations. The existence of inorganic material like clay in polymer matrix, generally, enhances the thermal stability of the nanocomposites. Moreover, clay as a refractory material possesses better thermal properties.
3. It is observed, that the damping properties for epoxy/UP system as a function of clay is increased appreciably at 4 wt. % clay content. Among all clay variations, 4 wt. % clay samples show better damping properties. This is due to the better dispersion of clay particles in the blend. Moreover, clay in said to have reasonably good vibration damping property.
4. Validation of the experimental results using Design Expert software, indicate negligible variations with the software generated results. This proves that the experimental results are correct and can be considered as best-fit values.
5. The epoxy/UP/clay nanocomposite with 3% clay content can be applied as superior, low-cost materials in the following areas: 1) As fuel tank in vehicles (presence of nanoclay reduces the transmission of solvents into polymer) 2) As fire-retardants and fire-resistant cables (the epoxy/UP/clay nanocomposite possesses better thermal stability and the variation of glass transition temperatures between 3 wt.% and 5 wt.% clay content is very negligible) 3) can be used to make superior, low-cost film material (as previous studies indicate that presence of nanoclay reduces haze in the film and improves transparency, in comparison to films made using conventional polymers) 4) automobile components and accessories like control panel boards, interior parts of automobile etc..and for many more applications.

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