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# The Production of Clay Nano-Composite Epoxy and Comparison of Its Properties with Epoxy Resins

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**ABSTRACT**

In this research, the layers separation and the intercalated nano-composites is studied with a new method using compressed air nozzles and the shear force in order to do lamination of layers within the epoxy matrix in a best way. Full separation of clay layers will cause the interaction with the surface area of the clay layers and the epoxy reaches its maximum. Then the mechanical properties such as tensile strength, elastic modulus, toughness and stiffness of the experimental conditions are discussed. Our results show that using method if combining nozzle with less diameter results the best in most cases and in general, use of 1 mm diameter nozzle and the amount of 5 wt% clay are the optimum conditions for maximum mechanical properties.

**KEY WORDS:** Nano composite, Epoxy resins, ultra sonic, mixing

## INTRODUCTION

Nano-composites reinforced with clay, are a group of advanced nano-composites with properties such as strength, high heat resistance, low weight and high thermal stability which are under particular interest.

Thickness of clay layers is about 1 nm and their perspective ratio is very high, so some weight percentage of clay which is dispersed in a matrix system correctly, compared to traditional composites, is creating more surface area for interactions between the filler and the matrix [1].

Many researches has been done in this field, thus it caused increases of mechanical properties such as strength and toughness of heat properties and leak with a minimum of filler.

The main problem for generating nano-composites is obtaining an effective dispersion of nano-clay in a polymer matrix [2, 3].

In previous studies, different methods have been used for cereal nano-composites. Such as in situ polymerization, Solid intercalation, the melting process, Co-vulcanization and Sol-gel method. It is obvious that using a method for creating complete lamination and good dispersion of clay particles in the polymer matrix is essential for improving properties of nano-composites [2, 4-6].

The use of nano-particles in the epoxy matrix systems has particular importance in the development of heat resistance composites [7].

In a research, an increase in the elastic modulus of nano-composites of epoxy / clay obtained by combining the shear strength and comparison with the direct synthesis method has been reported.

In some researches, the effects of the modified clay in elastic modulus and toughness, tensile strength, impact strength, thermal and mechanical properties and deformation mechanisms in epoxy matrix have been studied [1، 3, 8-10].

In a new study, the mechanical properties of nano-composite epoxy / clay produced in a centrifuge machine have been studied [11].

Given the above, the composition and manufacture of nano-composites should be done in a way that cause complete separation and good dispersion of clay particles in the matrix epoxy [12-13].

This combination should be done in such a way that interaction of middle phase between clay particles improves the mechanical properties of epoxy [14].

Since the layer thickness is on the nano-scale, using shear force can be a good way to segregate and distribute them within the matrix [15, 16]. Therefore, given the importance of the topic and international attention to the approach of nano-composites and unknown mechanism of breaking the material, the purpose of this research study is to understand the mechanical behavior and breaking mechanisms of nano-composites epoxy - clay.

## EXPERIMENTAL

### *Materials*

Used material in this research is included epoxy resin and modified clay (organ clay) in dimensions 25-35 nm. Components of used epoxy are epoxy resin liquid(diglycidyl ether of biphenyl A, DGEBA) with trade name Epian

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06 from Khuzestan petrochemical company and solidifier tri-ethylene thetra amine (TETA) from china country. Used clay is with epoxy grade and from products ATP Company. It is quotable that intended clay is purchased in modified form respective company and no treatment is done during production on it.

*Machines*

Used machines in this project are Ultra sonic mixture, X ray diffraction made in Philips company, tension, adhesion of Elcometer and industrial spray with different nozzles.

**Methods**

*Samples preparation*

Epoxy resin and clay with defined weight ratio (3, 5 and 7 weight percent) for 8 hour with Ultra sonic mixture are mixed and after adding solidifier the mixing is continued. In the end of mixture process the materials are transferred into the cast and for 16 hours in 120 $^{\circ}C$ are cocked. Making of pure epoxy samples is done with mechanical mixture.

*Samples recognition*

Clay distribution in epoxy with X ray diffraction and microscopic transient electron method is investigated. Pressure and tension behavior of epoxy-clay nano composites are respectively according to standard D638 and ASTM D695 and velocity of applied force are 1.5 and 5 mm/min and average results are reported for at least four measurements. Examination of fracture behavior and fracture toughness( $K_{IC}$ ) of prepared formula is measured with one-slot samples by 3 point-bending method according to standard ASTM D5045. After The preparation of samples, we poured them into the spray device and through initiation of the shear force created by the compressed air at the end of nozzle, were collected in the plastic container which was placed at the end of the nozzle, And this process for different weight percentage of clay (0, 3, 5 and 7) and for two different nozzle diameters (1 and 1.5 mm) was repeated.

To be assured from correctness of results, in each item 3samples are examined. Also in this research, a microscopic study of dynamism electron is done to recognize the mechanism of epoxy-clay nano composite fracture.

## RESULTS AND DISCUSSION

In the Figure 1 the x-ray diffraction diagram shows the morphology of the layered nano-composites containing 5 wt% and 7% clay. But to comment on the morphology of the samples in 3%, we need to repeat the test in lower angles which the corresponding diagram is shown in Figure 2.

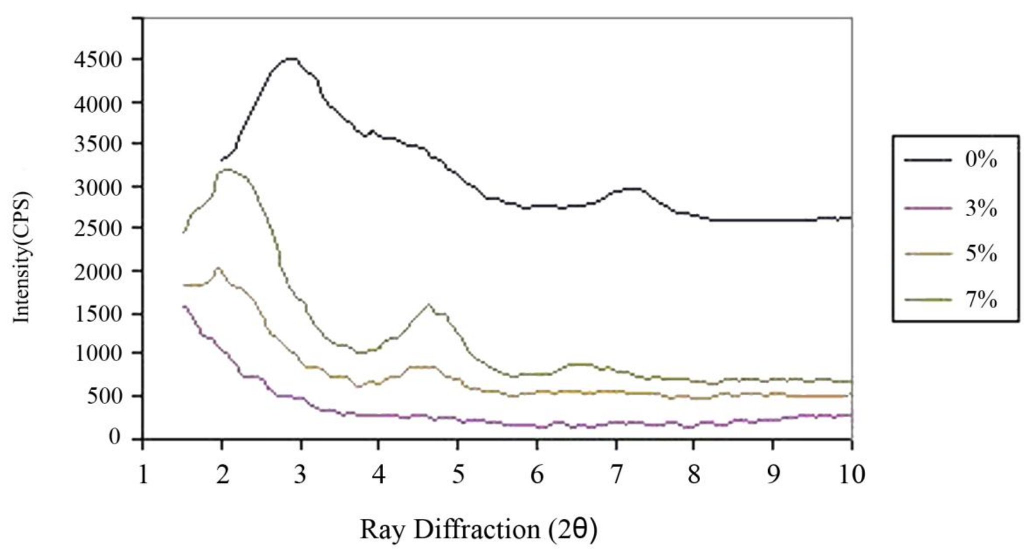


Fig. 1 - x-ray diffraction diagrams for nano-composites contain different amounts of clay

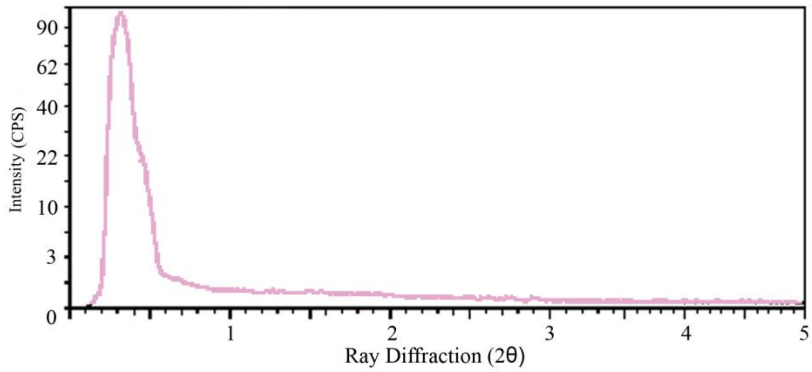


Fig. 2 - x-ray diffraction diagrams for nano-composites contain 3% clay

As is evident in Figure 1 and 2, the combination of 3% which is the lowest combined percentage of clay in the produced nano-composites, only one peak is observed at a very small angle. Thus, at this percentage of combination the structure has been broken. However, the morphology of the combined percentages of 5% and 7%, due to the presence of all three of these peaks at angles.

Table 1 shows the values of angles and distances (Intercalated) 2 to 10 degrees nano-composite structure resulting in an interlayer between layers of samples containing different amounts of clay.

Table 1: the values of angles and distances between layers of samples containing different amounts of clay

| **Sample Specifications** | **Angle of first peak (Degree)** | **Distances between layers (Angstrom)** |
|---|---|---|
| Containing 0% wt Clay | 2.75 | 31.5 |
| Containing 3% wt Clay | 0.324 | 272.718 |
| Containing 5% wt Clay | 2.050 | 43.059 |
| Containing 7% wt Clay | 1. 955 | 45.152 |

After that, by increase of clay to 5% wt, interlayer distance increase shows a significant reduction. But in the composition 7% the interlayer distances are increased again. Increase percentage of clay in polymer matrix, which is equivalent to a reduction in polymer percentage, On the one hand, reduces the possibility of polymer chains entrance to the distance between the layers and at the other hand increases of the dynamic viscosity as a direct function with increase of clay That creates a higher shear stress (shear stress = shear viscosity * velocity), helps to increase distance and separation of clay layers and these two factors are always in competition. Thus, it can be said that in the combination of 5% compared with 3% for the first factor was the dominant and in the composition of 7% to 5% increase in the dynamic viscosity is overcome.

After analyzing the results obtained in relation to mechanical properties such as tensile strength, elastic modulus, and toughness in terms of the Combination method (combining manual nozzle 1 and nozzle 2) with different weight percentages of clay were summarized in tables.

The relationship between the composition tensile strength and preparation method of nano-composite samples with clay content is in Figure 3.

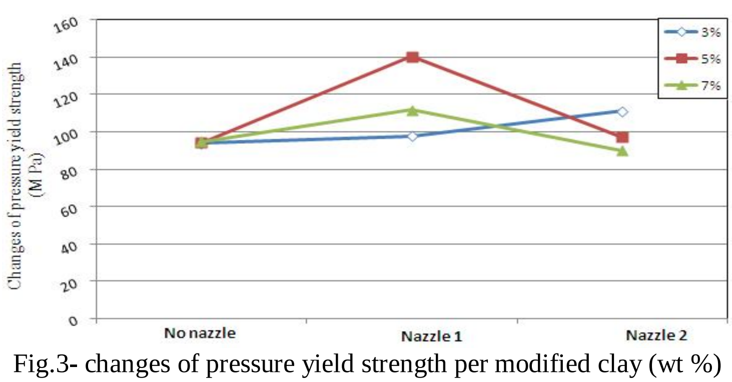


Fig.3- changes of pressure yield strength per modified clay (wt %)

It is noticeable that use of nozzles in combination epoxy / clay leads production of specimens with higher strength.

Also nozzle 1 in comparison by the nozzle 2 because of lower diameter has produced parts with greater strength. It causes the clay particles to be divided into smaller layers. Between the three different methods and different amounts of clay, that was obtained by combining the nozzle 1 and 5 wt% clay, clearly be seen more strong in comparison by other conditions.

Figure 4 shows relationship between combination methods and elastic modulus of prepared hub nano-composites.

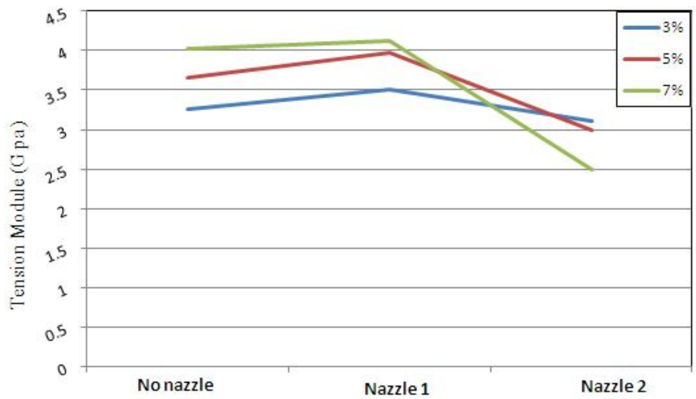


Fig.4- changes of tension module for nano composite per amount of modified clay (wt %)

Figure 4 shows changes of tension module in nano composite epoxy-clay per amount of modified clay in. According to tension test results, yaunge module has linear dependence with nano filler amount in polymer.

As it is clear from the diagram above, using a combination nozzle for achieving greater separation of the clay layers leads to production of flexible segments.

Also produced segments by the nozzle 1 are not different in elastic modulus with the manual combination method but nozzle 2 decreases the elastic modulus and we can say that using a combination of nozzle cause acquisitions of more flexible segments.

Figure 5 shows the toughness test results. As observed, epoxy nano composites have more toughness than pure epoxy and toughness of this kind of nano composite depends on nano filler amount in polymer.

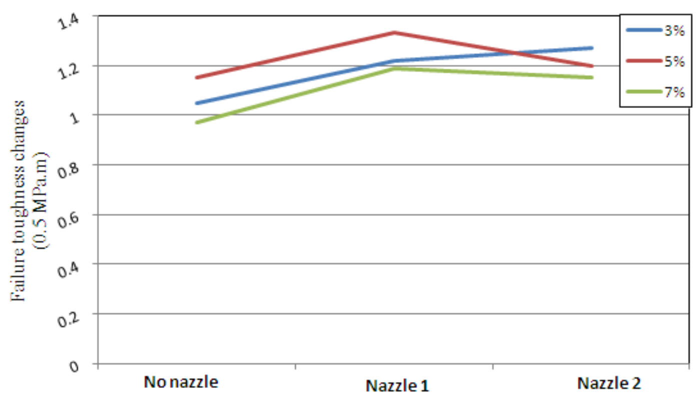


Fig. 5- Failure toughness changes of nano composite per modified clay (wt %)

Figure 6 microscopic air bubbles inside the nano-composite samples (bright spots), and Figure 7 also shows large air bubbles in the samples.

Fig.6-microscopic air bubbles inside the nano-composite

Fig.7- Shows large air bubbles in the samples

The presence air bubbles in the parts are the main problems in combining nozzle method.

The presence of air bubbles within the sample decreases considerably the mechanical properties and the existence of irregularities in the tensile test results.

Combining nozzle method causes an increase in composition of microscopic bubbles in the sample which finally these bubbles can decrease the mechanical properties.

But we can significantly increase the mechanical properties by careful mixing process of epoxy mixture and more precisely control of nano-composites production process and provide a way to prevent or reduce the possibility of the formation of bubbles.

Figure 8 shows microscope images of dynamism electron in the failure surface of pure epoxy and epoxy nano composite-3 weight percent clay. According to microscopic studies, a notable difference between failure surface of pure epoxy and epoxy-clay system is observed. Failure surface of pure epoxy is completely smooth and is similar to failure surface of fragile polymers. That represents too much resistance against crack development. In spite of pure epoxy, failure surface of epoxy nano composite is too rough.. In some areas it seems that there is an aggregation of clay layers. Crack transits among them and at process zone in failure surface lines similar continuation after agglomeration areas of clay is observed. In this figure image of failure surface for nano composite 3 weight percent clay as a sample is shown. Failure surface of other compounds are like 3 weight percent clay compounds. With this different that roughness of failure surface depends on clay amount in it. So if the amount of nano filler is increased the failure surface of nano composites are become rougher. Improvement of failure behavior in epoxy-clay nano composites rather to pure epoxy is related to crack deflection, formation of new surfaces in front and also failure of clay piles.

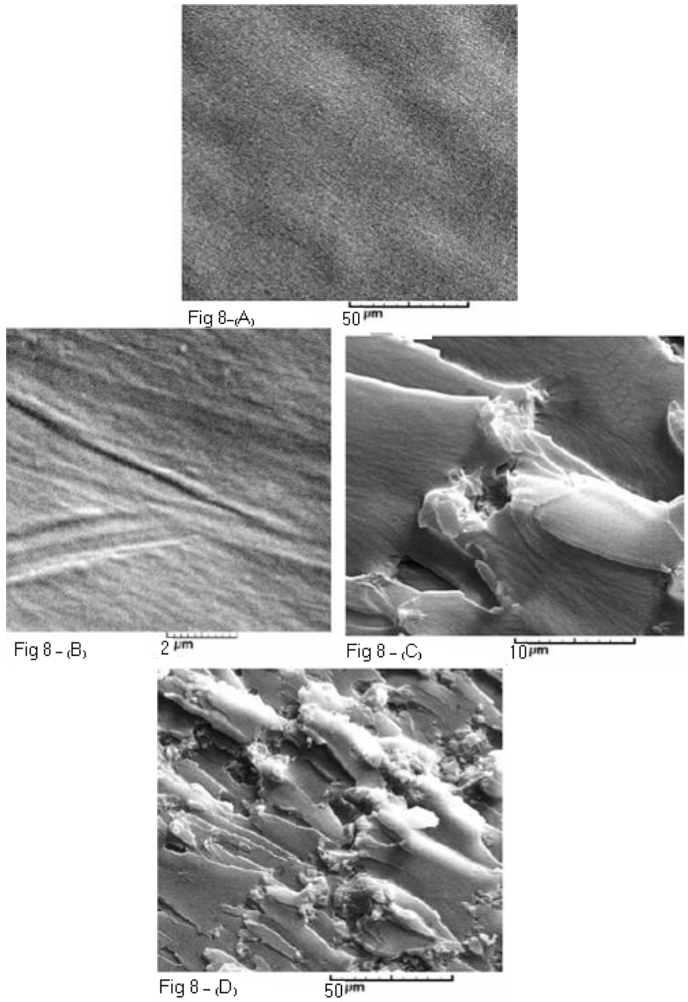


Fig. 8- Microscope image of dynamism electron in the failure surface : (A) (B) pure epoxy (C)(D) nano composite

## Conclusions

Examination of mechanical behavior and failure in epoxy-clay nano composite is shown that existence of modified clay in epoxy increases the adhesive yield of this material in high tension condition.

According to the tests and analyzing the results of the tensile test, these results will be obtained that using nozzle 1 and clay content of 5% by weight is the optimum combination of method and improves the various mechanical properties. It was also concluded that using nozzle will produce nano-composites with improved properties.

And a nozzle with a less diameter can result achievement of stronger, more flexible and tougher samples and better mechanical properties.

But since the clay nano-sheets has nanometer (nm) thickness and nozzle diameter is about 1 millimeter (mm) and about a million times larger, so it's not possible to separate all the layers clay.

Also formation of air bubbles in nozzle combination method decrease the obtained mechanical properties.

But we can significantly increase the mechanical properties by more precisely control of nano-composites production process and provide a way to prevent or reduce the possibility of the formation of bubbles.

Adding modified clay to resin epoxy improves coincidently solidness and toughness of this material. Microscopic considerations are shown that crack angle, formation of new surfaces and failure of new piles are some of the effective mechanisms to increase the toughness of epoxy-clay nano composite with intercalation shape.

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