High Functional Alumino-Silicate / Polymer Nanocomposite[†]

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Abstract

Towards Innovating of advanced clay based - polymer nanocomposite with improved properties, this research is proposed and conducted. This research project is a phase of big research theme towards overcoming the difficulties and shortcomings of the previous studies for each of the experimental work (mixing, processing) and fundamental analysis (characterization, modeling, mechanical analyses) to produce controlled nanocomposites with required properties. This research is devoted to fundamental study for predicting the aspects of nanoclay particles – polymer interaction during processing and final product. In this regards a new approach and model are applied for predicting and innovating modified mechanical properties for producing a new nanocomposite. Several types of both nanoclay and polymer materials are used. Design, modeling, processing, characterization, testing, fundamental analyses and mechanism are considered regarding the influences of the some other controlling parameters such as interfacial properties and thermal effect. The study depended mainly on the directional fracture energy approach.

KEY WORDS: (Silicate aluminum), (Nanocomposite), (High functional), (Polyethylene), (Directional fracture approach)

1. Introduction

The research was mainly prepared for investigating and exploring some aspects that are pertinent to clay based polymer nanocomposites as the conceptual approach, modeling, mechanism, analyses, characterization and prediction of defects and fracture. These objective are achieved through proposing of a new concept and understanding, proposing a new analytical approach (directional energy approach) for studying the problem, developing a new modeling and mechanism, prediction of cracks and fractures morphology and aspects which control the nanocomposite energy capacity and life time. The results of this research are important for each of industrial field of nanocomposite and academics. Nowadays, it attracts the attention of the first world in all fields industry and applications. On the academic level, the research fields for analyses and design of nanocomposites has a great importance which the results of this research can be good base for good research. The results open the door for using the domestic of polymers and clay for producing material nanocomposites with high quality and good mechanical properties. In the same time it will encourage the domestic industries to manufacture nanocomposites on commercial scale.

Due to natural properties of clay minerals as nano

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materials with high aspect ratio, large surface area, low cost rather than its friendship environmentally, clay platelets are very convenient for producing advanced nano-composite with unique properties by using it as filler material for reinforcing of another materials such as polymers to produce different materials called claybased polymer nano-composite. These types of nanocomposites have new different properties than the properties of original components which make it suitable for many different fields of industrial and commercial application such as automobiles, medicine, electronics, sensors, constructions, packaging, coating, drugs, biomedical engineering and others. The production of these nano- composite materials go through several steps of material preparation, mixing and processing. Basically, design of these nano-composites depends on simple concept and mechanism of strengthening of a material of weak properties by adding a small amount of anther material of strong properties. Reinforcing of main material (polymer) with a certain ratio (5%-15%) of the filler (nano clay), will change the polymer to another advanced functional material. The morphology, characterization and analysis of these materials are based on that mechanism. Several problems and difficulties faced the manufacturing of these nanocomposites. In current study, a new point of view for each of the concept, mechanism is introduced followed

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by new techniques for mixing and processing then new method of analysis and characterization. The study depends on the energy approach regarding each of the interfacial bond strength between the clay nano fibers and the polymer, voids and inclusions, de-bonding process between fibers and polymer, fiber orientation, fiber distribution and intensity, fracture, and cracks. The study uses the natural Saudi nano clay and polymers while previous studies used the synthesized clay. The study is trying to solve the problems and difficulties of clay/polymer nano-composite to improve its industries.

Previous works could not investigate the most important aspect of the clay based polymer nanocomposites¹⁻¹⁹). Therefore this research is conducted mainly for developing the clay based nanocomposite. Extensive studies have been made on this field but could not solve any of these problems. The factors governing the strength and the required properties on the nanocomposite are the original properties of the composite components such as mechanical properties, structure, and fracture properties, thermal and chemical properties. This research will include experimental and theoretical works. Experimental work will include processing, characterization and testing while the theoretical work includes concept, approach, modeling, mechanism, analyses and comparisons. The directional strain energy approach will be applied regarding the energy components. It will consider size, and shape effects. The study will be carried out on two or three types of clays and polymers.

2. Experimental

2.1. Fundamentals procedures of the approach

For experimental model and work in addition to theoretical analyses, the following procedures and methodology should be carried out.

2.1.1. Material selection and preparation

Material selection should be made carefully for both of nano clay and polymer matrix. In current study the materials are Montimorillonite MMT clay, Figs. 1, 2, 6, while domestic Saudi polymers such as Polyethylene PE produced by SABIC, Figs. 3, 7. Aspect ratio and surface area of nano clay platelets are effective factors for producing the bond. The most important step in the preparation is materials compatibility. Good compatibility between clay and polymer should be made. This is because the polymer and clay are completely different. Polymer is organic and hydrophobic material while nano clay is inorganic and hydrophilic rather than polymer is ductile thermoplastic material while clay is brittle thermosetting. Clay surface should be treated to be organic using organic compatible material such as maleic anhydride polyethylene for surface modification of MMT to be compatible with PP to facilitate interfacial bond. After that, the materials will be ready for mixing and processing stages.



Fig. 1 Modified Organic Nano clay (nanomer 1.3 produced by Nanocore).



Fig. 2 Modified Organic Nano clay (nanomer 1.3).



Fig. 3 Saudi Arabian polymer.



Fig. 4 American clay/polymer Nanocomposite.



Fig. 5 Current Saudi clay/polymer Nanocomposite produced by the author.



Fig. 6 Chemical analysis EDAX for Nanomer 1.3.

2.1.2. Mixing, extruding and processing

After preparation of nano clay to be organic clay, it will be mixed with polymer using dry mix method for several times by ordinary mixer such as roll mill mixer until the homogeneity of the composite then the composite will be mixed again by using high shear mixer. This will facilitate the dispersing of clay fibers in polymer matrix, intercalation, exfoliation, and bond between clay and polymer rather than producing uniform and homogeneous distribution with minimum agglomeration, defects and voids. This mixing technique should be repeated for several times.

After mixing for several times, the composite will be extruded using systems of both single extruder and twin extruder with length-diameter ratio 40 and eight different zones of temperature including feed and die zones. These high shear extruders and heating will help in, mixing, exfoliation, distribution, and dispersion of clay in polymer. Extruder characteristics such as diameter, length-diameter ratio, angle of the teeth, feed speed and rate, extruder speed and time of extruding and vacuum will control the processing. These conditions will control the production, the characteristics of bond, dispersion and exfoliation producing uniform material. The final produced nano composite after extruding will be prepared in the form of pellets.

The method of exfoliation of nano clay layers was made chemically by swelling as using chemical agent. The second method of exfoliation is mechanically by using mechanical mix. The high shear can help in dispersion of the nano clay in the polymer and vice versa mechanically but the chemical method is still better because it can be considered as self dispersion and self exfoliation special when using the natural materials. The third method (current method) uses the two methods together in the same time.

Several mixes of clay/polymers should be done using several ratios of nano clay powders such as (5%-15%) of polymer. Based on the applied analytical theory²⁰⁻²³⁾, well characterization, testing and analysis of the product should be done. Using SEM, EDAX and XRD techniques and testing for cracks, interfacial de-bonding, particle agglomeration, voids; checking for fracture toughness and fracture properties of the final material should be done as basic measurements for the nano particles composite quality. Nano distribution, orientation, exfoliation, dispersion and agglomeration should be evaluated.

The applied theory for the analysis is based on directional fracture approach DFA using dilatational – distortional strain energy theory. While nano particles are better not to be studied by continuum theory, the nano-composite actually in practice will behave in continuum approach behavior with consideration of presence of voids, cracks, fibers, etc. Therefore, the applied theory should follow the basics of the continuum approach. The theory is dilatational – distortional strain energy theory (MN-theory) based on directional fracture approach DFA²⁰⁻²³. From the mechanical point of view,

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voids, cracks, defects, de-bonding at interfaces between fibers and polymers will be produced during processing. The applied theory can deal with this material for investigating the defects and properties. Then, it will assist in improving the production performance, control the material production and prevention of each of the defects, cracks and voids. The analysis should be done for the actual material which includes cracks, defects, voids, inclusions. The polymer itself may contain voids which are full of gases or air due to gases or absence of good vacuum. Voids will produce stress concentration and cracks, then propagation to be large cracks which can produce failure or reducing of the capacity and lifetime. Some nano particles of filler reinforcements may produce inclusions due to the agglomeration of the nano particles making micro or macro particle making problems in the nano composite. If there is de-bond or weak bond at bi-interface between polymer and clay, it will produce cracks, then failures. Since there is no control on the material structure, it is very difficult to deal with clay/polymer nano composite as a regular composite with regular mechanical behavior such as unidirectional material, orthotropic material or anisotropic material. The clay/polymer nanocomposite of uniform distributed nano clay particles without agglomeration can be considered as isotropic composite. Therefore, as a general approach, the application of the theory will consider the nano-composite as isotropic material.

2.2. Mechanical and fracture testing

2.2.1. Testing of mechanical properties

The tests are made for investigating the mechanical properties such as the tensile strength, modulus of elasticity, elongation, plasticity, and stress-strain relation. Some samples are prepared for the tensile strength testing of plain samples without cracks to check the strength using displacement control machine as shown in **Fig. 9**. The results are shown in the **Figs. 10-12**, **Table 1**.

 Table 1 Mechanical properties of un-cracked.





Fig. 7 Chemical analysis EDAX for HDPE.



Fig. 8 Chemical analysis EDAX for clay/polymer nanocomposite.



Fig. 9 Clay/polymer nanocomposite sample during mechanical test.



Fig. 10 Critical stress intensity factor envelop (K_I-K_{II}) for Clay/polymer nanocomposite for Mixed mode edge cracks.

2.2.2. Fracture properties testing

The mechanical tests are made for the produced nanocomposite to investigate the fracture properties such as fracture toughness (critical stress intensity factor, K_{LC} = σ (π a) ^{0.5}, a = half crack length = edge crack length = 1.0 mm, σ = remote applied stress). fracture path and fracture surface. The tests are made at KACST in Saudi Arabia. Another clay/polymer nanocomposite samples of pre-made notches (Precracks) are investigated to predict experimentally the fracture toughness of mode I crack (critical stress intensity factor K_{IC}) as shown in Fig. 10. The tests are made under room temperature. The results are shown in Figs. 10-12, Table 1. The samples dimensions are 110 mm length, 12 mm width and thickness of about 1.5-2.0 mm. The pre-cracked samples prepared for testing the fracture, the cracks are double edge cracks of length about 1.0 mm at each edge. The cracks are located at the middle of the sample length between the grips of the machine.

3. Characterization

The characterization stage included mainly the microscopy analyses by each of SEM in addition to the crystalline analyses by X-ray diffraction analyses and chemical analyses of the materials by EDAX, **Figs. 6, 7, 8**. The characterization included the mechanical analyses test for mechanical properties and fracture properties.

The characterization includes the analytical microscopy investigation for each of clay, polymer and clay/polymer nanocomposite by using SEM, EDAX and XRD analyses. The nano structure and microstructure of each of clay, polymer and clay/polymer nanocomposites are investigated by SEM. The chemical analyses for each of clay, polymer and clay/polymer nanocomposite are investigated. The crystallinity of the clay particles was investigated by using XRD. SEM, XRD and tests analyses are made in King Saud University and KACST. The SEM analyses for each of clay, polymer and

Nanocomposite included the steps of samples preparation, characterization, nanostructure, morphology, defects and pores, fracture and cracks. Characterization included clay/polymer nanocomposite made in USA. A comparison is made between our produced nanocomposite in Saudi Arabia and USA Nanocomposite using SEM, **Figs. 4**, **5** and EDAX analyses, Fig. 6, 7, 8. The comparison included the microstructure, nano structure, morphology, defects, cracks, clay distribution, homogeneity, dispersion, intercalation, exfoliation and chemical analyses.

3.1. Characterization using SEM

3.1.1. Clay material

The filler material (clay) of the nanocomposite is characterized using field emission scanning electron microscope SEM, FEI type. The different types of nano clay are characterized to investigate each of the structure, morphology, dimensions, defects, distribution, pores, cracks, geometry and shape, agglomeration, exfoliation, nanostructure, particle size and deformations. The characterization included the different types of nano clay materials which include nanomer 1.3, Nanomer 1.34 and Nanomer I.22E produced by Nanocor USA. Samples of each type of clay powder is put in the small holder of SEM using double sided carbon tape for fixing it on the holder. Then the samples are coated by gold with thin layer to prevent the charging of the electron beam on the sample during the investigation. After coating of the samples by gold using plasma sputtering device, the samples are installed in the SEM chamber and became ready for investigation and analysis.

The results are shown in the Figs. 1-5 for each of the all types of clay minerals of MMT Nanomer 1.3, Nanomer 1.34, and Nanomer I.22E. From the figures, it is clear for all types of clay that the dimensions and size of the particles is same which are MMT clay mineral of thickness 1.0 nm, width of about 200-400 nm. The shape is almost rectangular for the flat particles. The clay dry particles are agglomerated together in micro particles of size about 20 microns and less. Some particles are separated as nano particles as an exfoliated particles.

3.1.2. Polymer

Three types of polymers are used in this research. There are three types; one of LDPE in the shape of pellets and two other types of HDPE in the shape of powder. The LDPE is amorphous material with short chains while the HDPE are crystalline materials with long chains. It is known that LDPE has short chains and weak mechanical properties while HDPE has long chains and good mechanical properties. Each of LDPE and HDPE are characterized by SEM to investigate the morphology, structure, defects, cracks, etc. the samples are gold coated. Figures 3, 4, 5, show the PE. It is clear the difference between the two types of amorphous and crystalline polymers. Each of them is related to the polyolefin family of the polymers.



Fig. 11 Load-displacement relation of un-cracked nanocomposite.



Fig. 12 Load-displacement relation of cracked nanocomposite.

3.1.3. Nano clay/ Polymer nanocomposite

The mix of nanoclay polymer composite is characterized by SEM. The structure of the nanocomposite, morphology of the components, distribution of nano clay, homogeneity, defects, cracks, orientation of the nano clay are characterized. Figures 4, 5 show the SEM characterization of clay / PE nanocomposites. The figures indicate the aspects of characterizations of the nanocomposites.

4. Test results (Tension test for uncracked and cracked samples)

It is shown from the tables, figures and photos the results of the experimental work of testing of cracked and uncracked samples of each of: 1. Pure HDPE, 2. Pure HDPE, 3. Pure LDPE, 4-Clay/polymer Nanocomposite -1, 5. Clay/Polymer nanocomposite -2, 6. Clay / polymer nanocomposite -3, 7. Clay / polymer nanocomposite -4, 8. Clay / polymer nanocomposite -5. Each type include 10 samples including Samples without cracks, Mode I crack, mode II crack and Mixed mode cracks for edge and central crack types. The test included The Fracture load (σ_{cr}), Fracture angle (θ_{cr}), Fracture toughness K_{I c} or critical stress intensity factor (K_I, K_{II}, K_I-K_{II}). It also included Elongation, Modulus of Elasticity, and tensile strength. The following examples are indicated:

4.1 Pure polymer (HDPE):

4.1.1. Without cracks: Thickness t = 2.0 mm, Width w = 12 mm, Tensile strength, $f_t = 33.423$ Mpa Elongation = 13.181 %, E = 1102.5 Mpa P max (maximum load) = critical load = 731 N Maximum displacement = 9 mm, Critical stress $\sigma_{cr} = 33.423$ Mpa

4.1.2. With mixed mode edge crack:

Crack length = 2.0 mm, β (crack angle) = 30, θ_{cr} (fracture angle) = 60, $K_{Ic} = \sigma_{cr} (\pi a)^{0.5} \sin \beta = 14 (N/mm^2) (mm)^{0.5}$, $K_{IIc} = \sigma_{cr} (\pi a)^{0.5} \cos \beta = 24.24 (N/mm^2) (mm)^{0.5}$, $\sigma_{cr} = 11.361 Mpa$

4.1.3. Edge crack mode I:

 $\begin{array}{l} Crack \ length = 2.0 \ mm, \ \beta \ (crack \ angle) = 90, \\ \theta_{cr} \ (fracture \ angle) = 0 \\ K_{Ic} = \sigma_{cr} \ (\pi a)^{0.5} \ sin \ \beta = 43.75 \ (N/mm^2) \ (mm)^{0.5} \\ K_{IIc} = \sigma_{cr} \ (\pi a)^{0.5} \ cos \ \beta = 0 (N/mm^2) \ (mm)^{0.5} \\ \sigma_{cr} = 17.5 \qquad Mpa, \qquad E = 1034 \ Mpa \\ P \ max \ (maximum \ load) = critical \ load = 363 \ N \end{array}$

4.1.4. With central crack mode I:

Crack length = 2.0 mm, β (crack angle) = 90, θ_{cr} (fracture angle) = 0 $K_{Ic} = \sigma_{cr} (\pi a)^{0.5} \sin \beta = 45 (N/mm^2) (mm)^{0.5}$ $K_{IIc} = \sigma_{cr} (\pi a)^{0.5} \cos \beta = 0 (N/mm^2) (mm)^{0.5}$ $\sigma_{cr} = 18$ Mpa, E = 1034 Mpa P max (maximum load) = critical load = 301 N

4.1.5. Mode II crack:

 $\begin{array}{l} Crack \ length = 2.0 \ mm, \ \beta \ (crack \ angle) = 0, \\ \theta_{cr} \ (fracture \ angle) = 88 \\ K_{Ic} = \sigma_{cr} \ (\pi a)^{0.5} \ sin \ \beta = 0 \ (N/mm^2) \ (mm)^{0.5} \\ K_{IIc} = \sigma_{cr} \ (\pi a)^{0.5} \ cos \ \beta = 22.5 \ (N/mm^2) \ (mm)^{0.5} \\ \sigma_{cr} = \tau \ _{cr} = 17.5 \qquad Mpa, \ E = 555 \qquad Mpa \\ P \ max \ (maximum \ load) = critical \ load = 22 \ N \end{array}$

5. Discussion of results

Nano clay (Montimorillonite MMT) as filler material and polymer (Polyethylene PE) as matrix are used for producing advanced material of nanocomposite (Montimorillonite-Polyethylene MMT-PE). The MMT nano clay as a filler or reinforcement has the advantages of large surface area (large surface area to mass ratio 800 m²/gm comparing to traditional filler material such as conventional clay particles or Talc powder), enhancement of each of mechanical, fracture, thermal, gas barrier, impact properties and others. It can realize these properties at small load. The major difficulties which can be found include the problems of realizing of each of the exfoliation, interfacial bond and compatibility of MMT particles and PP. Exfoliation is one of the terms used to determine the extent of polymer of clay in the dispersion matrix. Montmorillonite MMT is semectite clay with the structure consisting of two tetrahedral layers of silica sandwiching one octahedral layer of aluminum. Nano clay is hydrophilic, inorganic, thermosetting brittle material while polymer is hydrophobic, organic and thermoplastic material. Therefore, it was essential to use a third material such as Maleic anhydride polyethylene, for producing compatibility and bonding between these two different types of materials in addition to realizing homogeneity by making the filler clay powder compatible with the polymer matrix. The properties and mechanism of each of the polymer and clay are different and of course the clay-polymer nano composite will produce another material with new properties. The compatible material will make surface treatment for the clay particle to be organophilic material which can be bonded to the polymer in strong chemical bond producing material with good mechanical properties since it will reduce the cracks and enhance the fracture properties. If the compatible agent could not produce this condition, the properties of pure polymer will be better than the nano composite properties. If compatible agent could not cover all clay particles, defects will be created.

It is found that most important aspects for manufacturing of clay-polymer nano composites which affect the properties of the composite are homogeneous distribution of nano clay in the polymer matrix, clay powder size, clay powder shape, aspect ratio, surface area, agglomeration of clay powder, dispersion of clay in polymer matrix, exfoliation of clay particles, dispersion of polymer between clay particles, bond between clay and polymer, compatible agent, presence of defects, voids, inclusions, cracks, temperature, method of mixing, shear effect during mixing, method of extruding, extruder screw, extruding time and clay/polymer ratio. Orientation of nano clay fibers affects the properties greatly. Shear mix helps dispersion of clay in the polymer, mechanical exfoliation of clay and intercalation of polymer in clay layers but it may break fibers, break bonding and change the orientation. We have our new technique for mixing. The previous studies had no evidence or checking method for exfoliation of nano clay applied otherwise XRD analyses. There was no evidence on the distribution otherwise SEM or TEM. There was no evidence about the bond between fibers and polymers. Therefore, our approach depended on the mechanism and fracture mechanics which can give good record for assessment of these aspects and quality of the product by checking the fracture properties and cracks in addition to the other characterization methods.

As introduced in the research experimentally and theoretically, the research includes theoretical and experimental work. Each of the theoretical and experimental work based on new concept and approach which are depending on facts on the nanocomposites of clay / polymer mechanical behavior during the processing steps of the material. The experimental and theoretical works are followed by fundamental characterization and mechanical testing. The characterization included the microscopic analyses by SEM, crystallinity analyses by XRD, chemical analyses by EDAX, mechanical properties and fracture properties using Instron universal testing machine. The materials included different types of MMT nano clay. The polymer materials included three types of PE produced by SABIC. The polymers included one type of LDPE in the shape of pellets and two types of HDPE in the shape of powders. The filler materials included several types of MMT. The nano clay is organic clay by the surface modification with organic additive surfactant to the clay for making the clay compatible with the polymers in order to facilitate the bonding between the polymers and clays through making good cross linking between the polymer chains and the chains of the organic surfactant of the clay. This compatible organic clay contains the nano particles of the clay hidden in the organic surfactant which will react with the main material of polymer matrix. The results of beach of the research step were previously expected based on the proposed concept and approach. The results are satisfactory and matching with the objectives which include modeling, mechanism, concept, approach, characterization, morphology, cracks and fracture and mechanical properties.

The results are completely matching the expected deliverables and outcomes of the research which are a new analytical approach, a new modeling, a new mechanism considering the interfacial problems, and prediction of cracks and fracture aspects. Therefore, the pre-expected benefits of this research could be achieved as it was expecting.

6. Conclusion

As explained and discussed, the clay/polymer nano composite had difficulties and problems regarding each of the approach, production techniques and properties. Current research tried to find solutions for these difficulties. Current research depended on new concept and methodology based on the fracture mechanics. Therefore, applying the basics of directional fracture approach for understanding of nano composite structure makes a new step towards overcoming the difficulties and problems of the nano composite manufacturing. New technique for both of mixing and processing is developed. Another important point in this research is using the natural nano clay for producing of clay/polymer nano-composites. Using natural nano clay will save money, time and efforts for producing high quality materials. Using Domestic Saudi Arabian materials is one of the main targets rather than finding the solutions for the difficulties and problems of the previous researches.

The target of this research mainly was made first to produce nanocomposite with high quality based on new understanding and approach from the mechanical point of view since the previous studies still have difficulties to produce complete clay/polymer nanocomposite material. Therefore, the research depended firstly on investigating and finding out the problems and difficulties of the previous studies. It was found that the previous researches depended on just the chemical approach for producing the nanocomposite neglecting in the same time or ignoring the effect of mechanical behavior and mechanism of these complicated materials and the effects during the processing steps. Therefore, this research is conducted taking into consideration the recent advances of the concept and analyses for the composites and nanocomposites. The research depended on the directional fracture mechanics approach. Through the research steps and processing, the obtained results are fully satisfactory with the objectives. The results included a new analytical approach (directional energy approach) for studying the problem, a new modeling, a new mechanism considering the interfacial problems, prediction of cracks and fractures morphology where the objectives are modeling, simulation and mechanism, new concept and understanding, morphological aspects, and characterization, prediction of cracks and fractures and aspects which morphology control the nanocomposite energy capacity and life time, Mechanical analyses developing a new methodology for modeling and mechanism, conceptual approach and prediction of defects and fracture, and proposing a new analytical approach (directional energy approach) for studying the problem.

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