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Nano clay reinforced epoxy floor coating composites: preparation and thermo physical characterization

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Dedication

To the one who is worth praising and thanking........

The one who revives the skies by His throne and decorates the universe by His mention........

Allah

To our great prophet Mohammad and his posterity (Peace and Blessings

of Allah be upon him and them)....

To The man who taught me everything and supported me in this life.....

My dear father

The one who is my angel on earth..........

The air I breath and the pulse that beats in my chest.......

My second heart mother

To The whispers of flowers and fragrance of morning.......

The shoulder of tenderness and miracle of all time

My brother and sisters

To The dear of my heart and my life and my lovers

The person who did not lose sight of me or think......

To the one who encouraged me and made life beautiful in my eyes

Dear husband

The persone who gave me happiness and sadness at the same time I present this research with my regards....

شكر وتقدير

في البداية نحمد الله عز وجل الذي وفقنا في اتمام هذا البحث العلمي :

فالحمد لله حمدا كثيرا .

و بعد الحمد تتقدم بجزيل الشكر وعظيم الامتنان الى استاذنا الجليل الذي علمنا كيف نخوض معترك البحث العلمي الجاد الدكتور المشرف " عمار كاظم " الذي تفضل بالإشراف على هذا البحث, حيث قدم لنا كل النصح والارشاد طيلة فترة الاعداد فله منا كل الشكر والتقدير .

كما لا يفوتنا أن نتقدم بجزيل الشكر والعرفان الى اعضاء لجنة المناقشة الموقرة.

كما نتقدم بالشكر الجزيل لأساتذة قسم البوليمر ونخص بالذكر رئيس القسم

"الدكتور ذوالفقار كريم مزعل " والدكتور البروفيسور المرحوم "جليل كريم احمد

وكل من افادنا وساعدنا خلال مراحل الدراسة ...

شكرا لكم

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The Aim of Project

Preparation of a composite material of epoxy coating reinforced with Nano clays, study of its tribological and mechanical properties and its adhesion properties to the concrete floor layer.

Abstract

Floor coating is one of the modern methods, or it is one of the ways to replace common floors such as (ceramic, cashew, alabaster, etc.) with epoxy paint. The properties of epoxy floor coatings were studied in terms of mechanical properties, wear resistance, and tribological properties. Where nanoclays (MMT for epoxy) were added for reinforcement in different proportions [0, 0.5,1,2 and 4%] using the Ultrasonic dispersing device and the results showed that the wear resistance of the nanocoposites coating reinforced with 2% nano clay was improved by 82 % compared to pure epoxy. The hardness was increased by 13.8% compared to pure epoxy while the adhesive strength decreased with increasing the weight percentage of nanoclay in the composites.

CHAPTER ONE

General Introduction

1.1 Introduction

Epoxy is the family of basic components or cured end products of epoxy resins. Epoxy resins, also known as polyepoxides, are a class of reactive prepolymers and polymers which contain epoxide groups. The epoxide functional group is also collectively called epoxy The IUPAC name for an epoxide group is an oxirane [1].

The term "epoxy", "epoxy resin", or "epoxide" (Europe) refers to a broad group of reactive compounds that are characterized by the presence of an oxirane or epoxy ring as shown in the figure below[2].

Epoxy resins may be reacted (cross-linked) either with themselves through catalytic homopolymerisation, or with a wide range of co-reactants including polyfunctional amines, acids (and acid anhydrides), phenols, alcohols and thiols (usually called mercaptans). These co-reactants are often referred to as hardeners or curatives, and the cross-linking reaction is commonly referred to as curing [3].

Reaction of polyepoxides with themselves or with polyfunctional hardeners forms a thermosetting polymer, often with favorable mechanical properties and high thermal and chemical resistance. Epoxy has a wide range of applications, including metal coatings, composites use in electronics, electrical components (e.g. for chips on board), LEDs, high-tension electrical insulators, paint brush manufacturing, fiber-reinforced plastic materials, and adhesives for structural and other purpose [4].

The health risks associated with exposure to epoxy resin compounds include contact dermatitis and allergic reactions, as well as respiratory problems from breathing vapor and sanding dust, especially when not fully cured.[5]

1.2 Structure of Epoxy Resin

The epoxy groups at both terminals of the molecule and the hydroxyl groups at the midpoint of the molecule are highly reactive . The outstanding adhesion of epoxy resins is largely due to the secondary hydroxyl groups located along the molecular chain; the epoxy groups are generally consumed during cure[6] . The large part of the epoxy resin backbone contains aromatic rings, which provide a high degree of heat and chemical resistance[7]. The aliphatic sequences between ether linkages confer chemical resistance and flexibility [8].

The epoxy molecule can be of different molecular weight and chemistry. Resins can be low viscosity liquids or hard solids. Low viscosity can be obtained at 100% solids, which results in good penetration and wetting [9].A large variety of polymeric structures can be obtained depending on the polymerization reaction and the curing agents involved. This can lead to versatile resins that can cure slowly or very quickly at room or elevated temperatures [10].

No small molecules such as water are liberated during the curing process. Thus, epoxies exhibit low shrinkage, providing a very low degree of internal stress when cured [11].

Epoxy resins that are commercially produced are not necessarily completely linear or terminated with epoxy groups. Some degree of branching occurs, with the end groups being either epoxy or hydroxyl. The amount and degree of branching vary from resin to resin and from supplier to supplier. Epoxy resins are not completely difunctional. Tri-, tetra- and polyfunctionality are possible. Various end groups can be introduced as a consequence of the manufacturing process

1.3 Applications of Epoxy Resin

Epoxy resins are multipurpose construction materials and coating agents due to their excellent qualities, which include resistance to humidity and chemicals, good adherence to different substrates, and outstanding mechanical features [3].

Epoxy resins may be used in three major areas:

- Coatings
- Adhesives
- Construction and structural materials [12].

CHAPTER TWO

The Theoretical Part

2.1 Epoxy Coating Mean

An epoxy coating is a coating compound consisting of two distinct elements: an epoxy resin and a polyamine hardener (also known as a catalyst). When mixed, the resin and hardener engage in a chemical reaction that creates cross-linking of the elements as it cures. When the epoxy coating is fully cured, the resulting product is a durable, rigid plastic coating with numerous desirable mechanical properties [13].

2.2 Advantages of epoxy coating:

Epoxy coatings are renowned for their outstanding mechanical properties, such as hardness and durability, and abrasion, impact and chemical resistance. These attributes make epoxy coatings an ideal protective coating material for components in demanding industrial settings.

Epoxy floor coatings, for example, are commonly used to extend the life of concrete floors in industrial facilities, warehouses, logistic centers and other locations that are subjected to light vehicular traffic and medium to heavy foot traffic. Its resistance to attack from chemicals, such as those found in oils, cleaners and bleach, make epoxy coatings a popular protective medium in the automotive industry. Fusion bonded epoxy coating technology is used extensively in the oil & gas and water/wastewater industry to protect pipeline assets from corrosion [14].

2.3The types of coating by epoxy:

2.3.1LIQUID EPOXY COATINGS FOR ABOVE AND BELOW GROUND PIPELINES

Protal ARO™ is a VOC free, 100% solids liquid epoxy coating formulated for application over FBE as an abrasion resistant overlay coating (ARO). It is a 3:1 ratio coating that can be spray or hand applied in the shop or field. It can be used to protect FBE mainline coatings for directional drilling, bores, river crossings and other rough terrain applications. Protal ARO can also be used to provide additional protection to the coating on bends, fittings and fabrication when severe handling or rough terrain exists [15].

Figure-2.1- Epoxy coating for crude oil pipes

Protal ARO should not be applied directly to steel Protal 600 CTE Low VOC™ is a two-part high build coal tar epoxy that has low VOC. It can be applied as a single or two-coat system. It uses polyamide technology with excellent abrasion and chemical resistance. For long-term corrosion protection of steel and concrete substrates against water, wastewater, seawater, alkaline water and acidic water corrosion [16].

Protal 600 CTE Low VOC is designed to coat steel piles, sheet piles, lock gates, reservoirs, non-potable water pipelines, treatment / storage tanks, bridges and many other aggressive industrial applications [17].

2.3.2. Epoxy coating floor

Floors and epoxy coatings are becoming increasingly popular for use as commercial and industrial flooring. There are a number of different types of epoxy coatings that can be used on floors including self leveling epoxy coatings, self-dispersing epoxy coatings, mortar epoxy coatings, graveled epoxy coatings, epoxy terrazzo coatings, epoxy antistatic coatings, and vapor barrier epoxy coatings. Each of these types of epoxy coatings offers distinct advantages and disadvantages when compared to the others as shown in figure[2.4] [18].

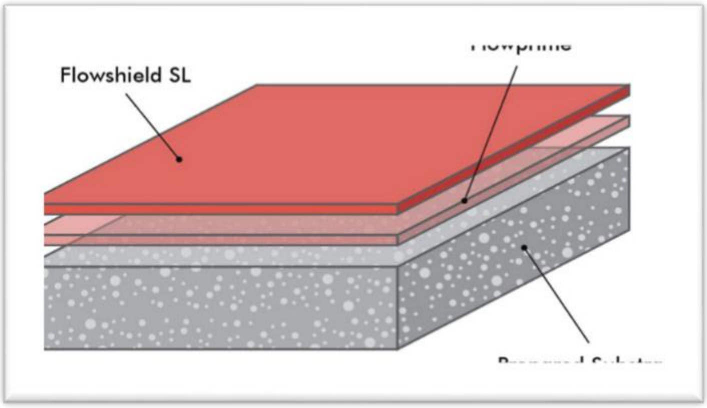


Figure 2.4 layers of epoxy coating for walls.

2.4. Epoxy Coating Choices for Flooring:

-Self Dispersing Epoxy Coatings for Floors: This type of epoxy coating is commonly used in areas that receive frequent forklift or heavy truck traffic as it has very good mechanical strength. Another type of this durable epoxy coating is self dispersing epoxy with quartz sand. This type of epoxy coating is commonly used in food processing industries or other locations where liquids are present because it has good anti-slip characteristics. [19]

-Self Leveling Epoxy Coatings for Floors: Self leveling epoxy coatings are easy to install over new and old concrete floors as they level easily creating a seamless and smooth surface. Self leveling epoxy coatings can be used in kitchens, dining rooms, storage places, garages, warehouses, office buildings, and more.

-Mortar Epoxy Coatings for Floors: This is the strongest of all epoxy floors. This type of epoxy coating is commonly used in heavy industry applications and can also be used to repair cracks before lying other types of epoxy floors.

-Graveled Epoxy Coatings for Floors: Graveled epoxy coatings are the most decorative epoxy flooring choice and they can be used for adding logotypes, brand marks, and decorative details to floors.

-Epoxy Terrazzo Floor Coatings: This type of epoxy flooring is very decorative and easy to clean. Epoxy terrazzo flooring is commonly used in large areas including hallways and entrances of commercial buildings, schools, and office buildings.

-Epoxy Antistatic Floor Coatings: This specialized epoxy coating is designed for use where static-sensitive electronic components are in permanent use and a static-free environment is most important such as in laboratories, hospitals, and electronics equipment manufacturing plants.

-Vapor Barrier Epoxy Coatings: Liquid epoxy vapor barriers are applied directly over concrete floors to provide an impenetrable surface that reduces vapor transmission to nearly zero. These epoxy coatings are typically applied prior to adding the final flooring surface including sheet vinyl, tile, carpet, or hardwood floors] 27].

• Epoxy Flaked Floor Coatings: This is not exactly a type of epoxy flooring, but rather a style of applying epoxy coatings. With this epoxy flooring technique, multi colored flakes or chips are added over the epoxy coating while it is still wet in order to provide a decorative finish or look [19].

Figure -2.5- coating floor [19]

From protecting pipelines to sealing warehouse floors, an epoxy surface coating protects surfaces strengthens materials, and protects them from corrosion and decay, making epoxy one of the most widely used industrial finishes. While epoxy coatings may initially cost more than other types of surface coating materials, its ability to provide superior and durable corrosion resistance, adhesion, and flexibility to a variety of surfaces ultimately make it a more economical choice, as the need to reapply the coating may be less frequent when materials are prepared well [20].

2.5 Types of Epoxy Surface Coating

There are a variety of applications for epoxy-based materials on the market, including coatings, adhesives, and the creation of composite materials. Those used for surface coatings are known for their good mechanical properties, electrical insulating properties, adhesion, and chemical- and heat resistance [21].

The most common types of epoxy resins include bisphenol A, bisphenol F, and phenolic novolac. Hardeners, or co-reactors, used with epoxy resins include polyamide, amidoamine, phenalkamine, aliphatic amine adducts, cycloaliphatic amine, aromatic amine, an aliphatic amine. The best resin and co-reactant combination depend on the purpose of the application and the performance requirements sought. If you seek a coating with high chemical and corrosion resistance, for instance, you may use a phenolic novolac resin with a polyamide co-reactant [22].

2.6 Epoxy Surface Coating Considerations

Epoxy coatings require specific environmental conditions to ensure the success of their application, as well as the proper preparation of the respective surfaces. When applying an epoxy coating to steel, for instance, you may need to remove the thin corrosion layer that naturally forms on the surface. Methods of cleaning surfaces include the use of chemicals or blasting products [23].

After you prepare and clean a surface, it becomes vulnerable to environmental contamination. When working with steel, for example, it takes as little as 30 minutes for flash rust to form [24]. The longer you wait to apply an epoxy surface coating, the shorter the expected field longevity becomes. For this reason, manufacturers provide specific instructions regarding how quickly to apply the first coating and the ideal environmental conditions [25].

In general, epoxy manufactures recommend the following conditions:

-Temperature: Above 55° F, or at least 5° F above the dew point, for the first 72 hours of drying; some coatings require thermal cures using high heat Relative humidity levels: 85 percent or less --Air: The area should have air circulating over the surface as it dries -Induction time: It is critical to allow the freshly mixed material to stand for the recommended amount of time, especially in humid or cold conditions

-Pot life: The amount of time the coating material remains suitable for use after mixing the resin and hardener; if you exceed the pot life, the coating may look usable but will perform poorly .

The success of a protective epoxy coating depends on the surface's cleaning and preparation, as well as the ambient conditions to which the surface is exposed during the preparation, application, and curing periods. When a controlled environment, such as a factory, isn't available, professionals look to temporary climate control solutions to create the ideal ambient conditions for every step of the epoxy surface coating process. From shipyards to power plants to construction sites, Polygon's custom temporary climate control technologies give you the ultimate control over ambient conditions, so your project is no longer at the mercy of the weather or HVAC systems. Contact Polygon to learn more about how its climate control solutions will enhance epoxy surface coatings and keep your project on schedule and within budget [26].

2.7 The Nano clay

Nano clay is a component composed of phyllosilicates, which are compounds based on the elements of oxygen, silicon, and other components and are degraded from natural sources and pretreated chemically. After further processing steps, Nano clay is used in different products. Due to their layered structure, they can swell or shrink as water accumulates or is withdrawn between layers. It is possible to increase the volume of Nano clay up to six times by water absorption and form stable gels. Nano clay has unique properties due to which, its uses can be found in different sectors such as to convert desert sand into fertile land. In this article, we'll see what is Nano clay and how it is used for different purpose [28].

2.8 Structure of Nano clay

Nano clay is composed of phyllosilicates which include groups of minerals such as talc (Mg3[Si4O10(OH)2]), Mica (KAl2[AlSi3O10(OH)2]), kaolin (Al2[Si2O5(OH)4]), montmorillonite (Mg0.33Al1.67[Si4O10(OH)2](Ca, Na) x (H2O)n), Serpentine (Mg3[Si2O5(OH)4]) or sepiolite (Mg4[Si6O15](OH)2 4H2O). Among other things, nanoclay differ in the size and sequence of the regions in which the SiO4 tetrahedra are oriented upwards or downwards in the layers. Additionally, nanoclay differ in the nature of the embedded ions. It has been shown that inhaling nanoclay particles causes only minimal and transient inflammation in the lungs. So far, there are no data on how nanoclay behave in the environment [29].

Montmorillonite, the most technically significant clay mineral as the main constituent of bentonite, is composed of SiO4 tetrahedron bilayers with deep-rooted octahedral layers of aluminum, iron, and hydroxide ions. A common montmorillonite particle comprises of approximately 1 nm thick aluminosilicate layers with lateral sizes in the series of 700 nm to about 10 µm, which accumulate into big stacks [30].

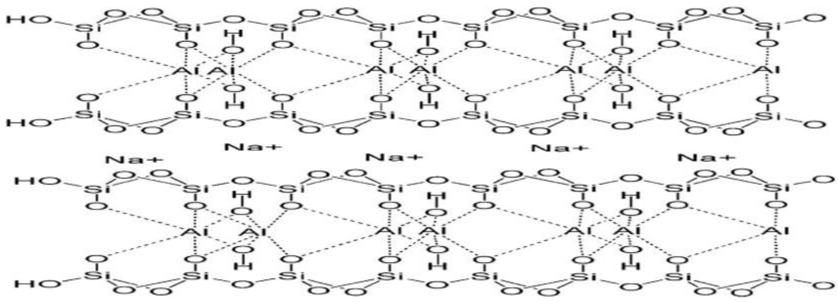


Figure 4 the structure of Nano clay

2.9 Applications of Nano clay

Nano clays have very important applications in different sectors. Some of the important ones are stated below:

1. They are not flammable; therefore, they are used in fire protection for plastics: Nanoclays are mixed in plastic, which decreases the quantity of combustible material. And when the plastic burns, it forms a layer that prevents the plastic from burning on .

2. Nanoclays are used as a plastic additive in food packaging films or beverage bottles (NOT in Europe). Plastic is thereby reinforced, less oxygen penetrates the film, so the food lasts longer or less carbon dioxide escapes the bottle. Nanoclay added plastics also have improved barrier and abrasion properties, higher tensile strength, low thermal expansion, superior surface qualities, and very good processing properties.

3.Nanoclays are also extensively used in paints.

4.Nanoclays can bind cesium and be employed for reducing theradioactive contamination.

5.To use as cat litter, a mixture of sepiolites, zeolites, bentonites or montmorillonites is used. They are characterized by a high absorption capability for water and odor-causing elements of cat feces and urine.

6.The use of phyllosilicates as fillers for polymer coatings in textiles is also being investigated.

7.The outstanding property profile of Nanoclays makes plastic nanoclay composites an exciting alternative to conventionally reinforced materials. In addition, nanoclay can be used to chemically mix incompatible plastics.

8.Nanoclay is not self-igniting, even as a finely divided mixture with air (dust) under the action of an ignition source. As nanoclay is not flammable, so there is no possibility for a dust explosion.

9. In combination with halogen-free mineral flame retardants, the addition of only 3% nanoclay to the plastic reduces the amount of aluminum trihydrate (ATH) required to pass the fire test by ten percent, while at the same time offering better flexibility and processing of the plastic.[31]

2.10 Previous Studies

Mohammad Reza et al, they studied the effect flooring grade epoxy/nanoSiO2 nanocomposites were prepared by in-situ polymerization method.Nano silica was treated by coupling agent in order to surface treating and introducing of reactive functional groups to achieving adequate bonding between polar inorganic nano particles and epoxy organic polymer. γ-Aminopropyltriethoxysilane (Amino A-100)was used as an effective and commercially available coupling agent and nano silica treated in acetone media.SEM observations of cured samples revealed that the nano silica was completely dispersed into polymer matrix into nanoscale particles. Thermal and physical properties of prepared samples were investigated and data showed improvements in physical and mechanical properties of the flooring samples in comparison with unfilled resin [33].

Fahriadi Pakayal et al., (2017) Studied the mechanical characteristics and thermal stability of thermoset epoxy as a function of room temperature vulcanization (RTV) silicone rubber content (5%, 10%, 15%, and 20% wt%). Thermoset epoxy with added RTV silicone rubber was subjected to testing and characterisation. Not only that, but RTV silicone rubber now has a lower tensile strength, elongation at break, and hardness. When 15% RTV silicone rubber was added, the best values for energy and impact strength were reached: 0.294 J and 6175 J/m², respectively. Additionally, thermoset epoxy's thermal stability may be enhanced by using RTV silicone rubber [34].

Ahmed J Farhan and Harith I Jaffer (2020) Prepared epoxy/RTV silicon rubber (SR) and unsaturated polyester/RTV silicon rubber (SR) blends are made by combining epoxy resins with SR and UPE resin with SR at weight percentages of 3, 5, 7, 10, and 20 respectively. This study aims to determine the mechanical characteristics of epoxy and unsaturated polyester as a function of RTV silicone rubber content (3, 5, 7, 10, and 20) wt%. Some of the mechanical parameters that were looked at were the impact strength, Shore D hardness, Young modulus, and flexural strength. The impact strength of the EP/SR and UPE/SR blends is maximized at 10%wt SR and 5 wt SR, respectively, according to the experimental data. In both EP/SR and UPE/SR, the hardness value was partially reduced as the SR component grew from 3%

to 20%wt. As the SR component grew from 3% to 20%wt in both EP/SR and UPE/SR, flexural strength quickly declined [35].

Nachiket G. Chanshetti1 and Anil S. Pol (2016) Examined the wear resistance and hardness of epoxy composites that have been supplemented with titanium dioxide (TiO2) and tungsten carbide (WC). There were three distinct volume proportions of TiO2 and WC put into the epoxy matrix: 5% ,and 10%. The specimens were made using the hand layup method, which included pouring epoxy and a combination of reinforcements into wooden molds. The tests were conducted in accordance with ASTM G99-95a and ASTM D 2240 standards for wear and (shore-D) hardness, respectively .Specimen wear resistance was evaluated using a pin-on-disk setup at different loads and rotational speeds. It was shown that both types of reinforcement in an epoxy matrix result in a decreased wear rate compared to neat epoxy. When compared to unfilled epoxy and 5% TiO2 filled epoxy ,as well as 10% TiO2 filled epoxy, the wear rate of the 10% TiO2 filled epoxy is reduced at all amounts of load. When it comes to WC filled epoxy composites, both 5% and 10% of WC filled epoxy composites will provide outcomes that are superior to unfilled epoxy resin in terms of decreased wear rate[36] .

Chapter Three

Experimental Part

3. Introduction

This chapter discusses all the experimental process and devises that used to obtained the experimental test, Hardness, Pull off, Wear and SEM test.

3.1 Materials

We have been supplied with materials for this project epoxy and nanoclay from the laboratories of the Polymers Department; the tests were also checked in the laboratories of the department**.**

Epoxy

PRODUCT INFORMATION

Packaging	2 kg (A+B)	P b
Colour	Yellowish/transparent	
Shelf life	12 months when unopened and stored	
Storage conditions	+5°C to +40°C	
Density	1.1 kg / ltr	
Viscosity	290 c. poise at 20°C R.T. type 130 c. poise at 30°C R.T. type	

Mixing Ratio	A:B = 2:1 (parts by weight and volume) Normal / R.T. type	
Pot Life	Temperature	2 kg R.T.
	40°C	15 min.
	30°C	30 min.
	20°C	60 min.

Nano clay

Characteristic properties	Cloisite® 15A	Cloisite® 30B	Cloisite® Na^+
Organic modifier	Dimethyl, dehydrogenated tallow, quaternary ammonium	Methyl, tallow, bis-2-hydroxyethyl, quaternary ammonium	None
Modifier concentration	125 meq/100 g clay	90 meq/100 g clay	None
% Moisture	<2	<2	4–9
% Weight loss on ignition	43	30	7
Typical dry particle sizes (microns)			
10% less than	2	2	2
50% less than	6	6	6
90% less than	13	13	13
Density			
Loose bulk, g/cc	10.79	14.25	12.45
Packed bulk, g/cc	18.64	22.71	20.95
Density, g/cc	1.66	1.98	2.86
Color	Off White	Off White	Off White
d_{001} (Å)	31.5	18.5	11.7

3.2The devices used

3.2.1 Hardness tester

A hardness test is required to measure the resistance of a material to indentation. A Shore D durometer instrument model (TIME 5431) made in China was used to test hardness samples. The specimen is circular in shape, as shown in figure 3.1[39].The test was done according to ASTM (D2240) [40]. The Shore device has a needle applied in a perpendicular direction to the sample. To obtain correct readings, the surface of the sample must be smooth and clean with a thickness of not less than 3 mm. Each specimen was tested six times at different positions on each specimen at the same time, and the final hardness is an average of them. Features of the Time TH210 Shore D Hardness Tester: pocket-sized Shore D Digital Hardness Tester with integrated probebright LCD displaydisplays hardness result, average and maximum hardness values peak value lock battery indicator measuring range of 0-100 HD 300 hours of operation with standard batteries automatic shut-off conforms to standards DIN 53505, ASTM D2240, ISO 7619, JIS K7215 manufacturer's certificate of conformity. optional 4,5 v AC/DC adaptor Figure (3.1).

Figure 3.1 Hardness device/ shore D

3.2.2 Pull-off Adhesion Test

A satisfactory pull-off test according to ASTM D-4541[41] is a common method for testing coating and adhesion quality; it entails adhering a dolly to a substrate (metal) with glue . Then, the dolly and the adhesive or coating are taken off the substrate by pulling perpendicular to the surface. Epoxy, epoxy mixtures, and WC/epoxy nanocomposite are used as coatings Figure (3.2) [42].

Figure 3.2 Pull-off samples adhasion

3.2.3. Wear test

The wear test was achieved according to ASTM G99-17 [43] using a pinon-disc machine U.S.A MT4003 version 10 micro-test. A polymer disc with dimensions of 40 millimeters in diameter and 4 millimeters in thickness was moving around a steel pin(16 Mn Cr5) with dimensions of 6 millimeters in diameter, a hardness of sphere of HRC 56, and a surface roughness of R=3.2 micrometers in a vertical configuration under a load of 30 newtons. At a speed of 300 rpm, a sliding distance of 235 m, and a time of 25 min, the pin was gliding along a track that was 24 millimeters in diameter. The wear specimen is shown in Figure (3.3).The test was done in the laboratory of Babylon University/College of Material Engineering/Department of Metallurgical. The investigation into the wear rate, which is shown below,uses the following relation

W.R =ΔW / S.D………………….. (3.1)

Where-:

W.R: The weighted measure of sliding wear is expressed as grams per millimeter.

The following relationship will be used to compute the change in weight (ΔW) that occurred during the experiment: -

ΔW=W1-W2……………………… (3.2)

Where-:

W1: Before the analysis, the sample's weight (g)

W2: The sample's final weight after being tested (g)

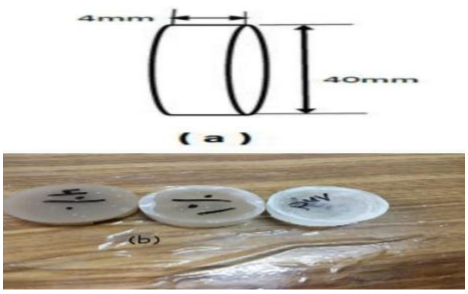


Figure 3. 3: (a) Aschematic of wear specimen, (b) wear test sample.

Figure (3.4) wear tester

S.D: a sliding distance that is determined from the lows that are presented below :

S.D=S*t…………………(3.3)

S: the rate of sliding (in millimeters per minute).

t: the time of test (min).

3.3. Sample Preparation

The surface has been prepared to obtain good adhesion and adhesion layer. At first, we cleaned the concrete surface, cleaned it with ethanol, dried it and applied a layer of masking tape with a thickness of 24 mm to obtain a coating layer of the same thickness as the masking tape layer. we added the nano-clay to the epoxy and mixed it using setters and added the hardener, and dispersed the mixture using ultrasonic waves, then we entered it into a vacuum system to remove bubbles. Next, the mixture is painted on the surface and left for 24 hours at room temperature, then we make curing at a temperature of 80 °C for an hour and at 120 °C for an hour too.

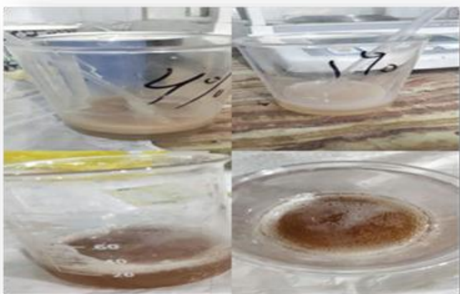


Figure (3.5) nanocomposit coating preparation

Results and discussion

Coefficient of Friction

It can be seen from Figure 10 that the friction coefficient of the pure epoxy sample was generally around 0.525, with increasing the nano clay percentage to the 2% percentage, the friction coefficient of the sample decreased to around (0.25) and this behavior due to the role of nanoclay that play as a solid lubricant. When the nano clay content reached to the 4%, the coefficient of friction increased to the 0.36, this due to increasing the percent of agglomerated portion of nanoclay in the epoxy matrix.

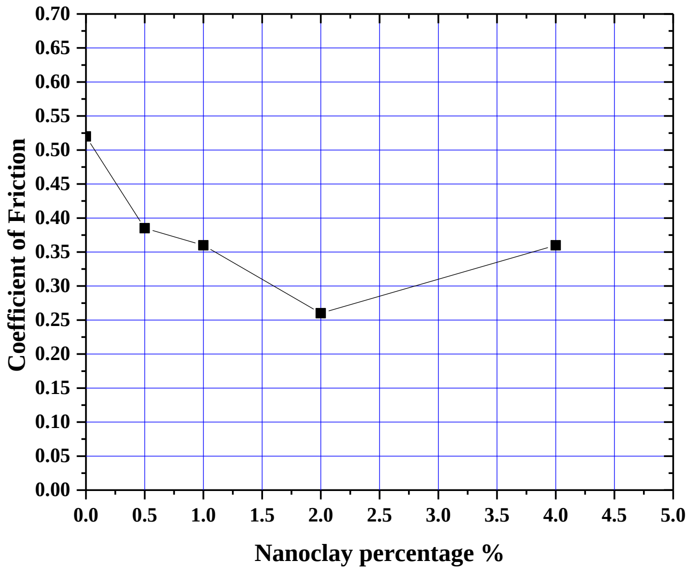


Figure 1 Friction test

Hardness

Fig. 4 shows the average hardness of different samples. By adding nanoclays into epoxy resin, the hardness of the composite increased proportionally with the nanoclay contents, where the hardness increased around 20% for epoxy reinforced with 4% nanoclay as a compare with pure epoxy coating. Hardness varies with the weight percentage of the filler material, which is to be expected because the nanoclays used have a higher hardness than the epoxy resin itself and good dispersion leads to an almost continuous increase in composite hardness.

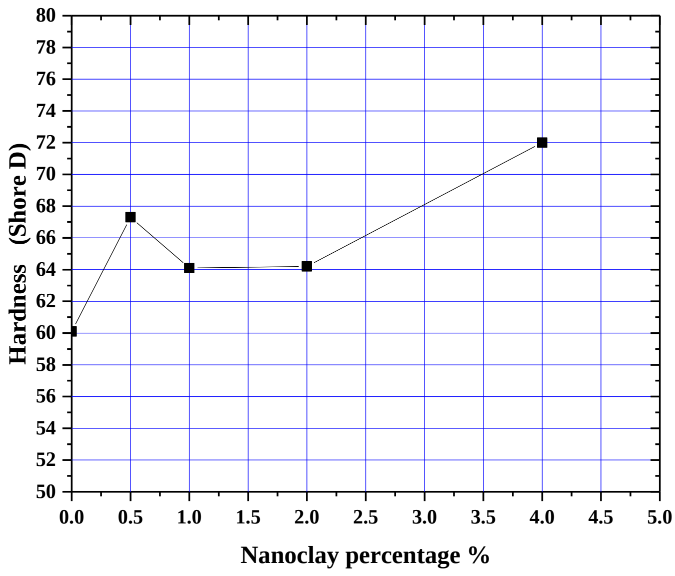


Figure 2 Shore D test

Pull off test

Adhesion testing has become a popular method of quantifying the strength of paints and coatings. One of the most popular methods for investigating adhesion strength is the quantitative pull-off test. A pull-off test compliant with ASTM D-4541 is one of the universal procedures used for quality coating checks; this involves a gluing the dolly to the concrete surface, then pulling the dolly using a force perpendicular to the surface to remove the dolly and the coating from the substrate.

The method includes an evaluation procedure, known as adhesion, which tests the strength of the pull-off for coatings on solid substrate concrete. Surface maintains intactness at a specifically prescribed force, resulting in either a pass or fail; the durability of the highest perpendicular strength of a specific surface area before the detachment of plug material can also be ascertained.

The results showed that the adhesive strength decreased with increased the weight percentages of nano clay, this behavior due to presences the clay at the interface between epoxy coating and concrete substrate.

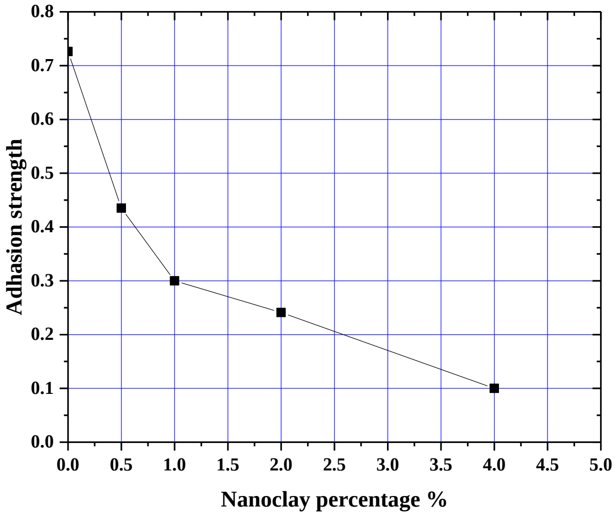
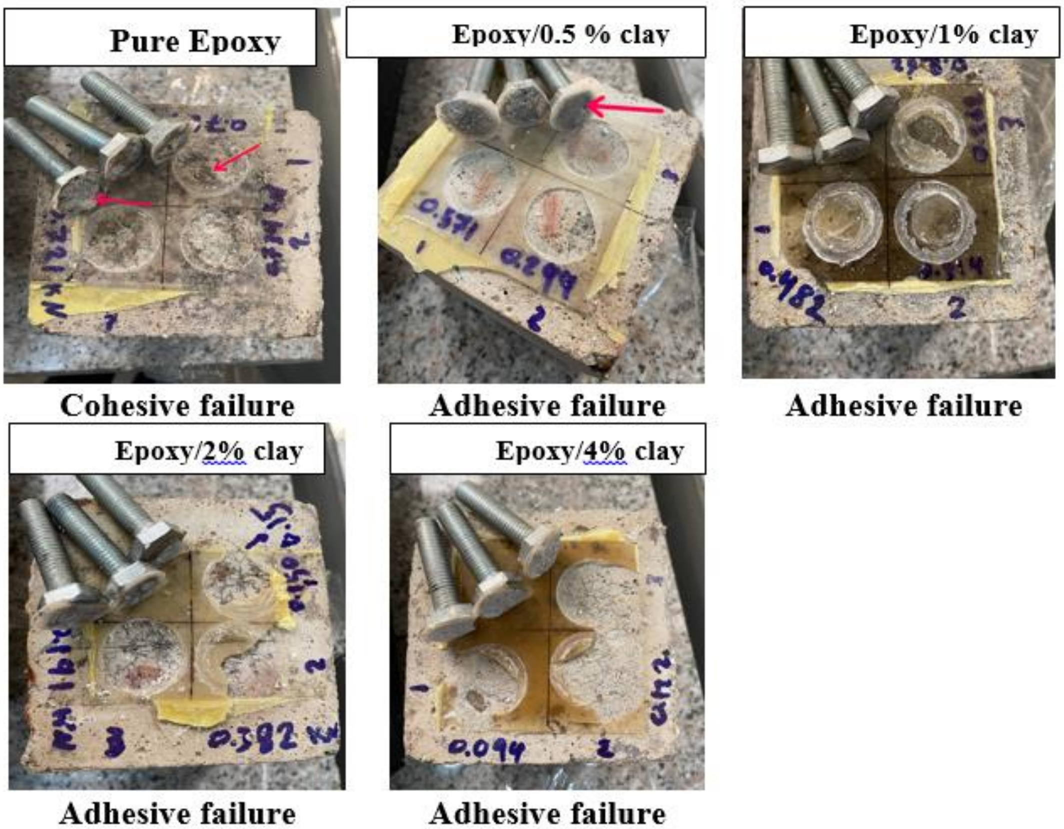


Figure 3 Pull off test

Failure in the pull- off test for adhesion can either be a result of concrete substrate failure, or a failure occurring between the layers of the particulate composite. In our cases, most failure modes were Adhesive failure between substrate and coating material.



Wear rate test

Wear resistance Fig. 4 shows the test results of the wear resistance of the pure epoxy and epoxy reinforced with different weight percentages of nanoclay. In general, the wear resistance increased with increasing the percentage of Nano clay, and the results showed that the sample with 2% nano clay weight percentage has the highest wear resistance. This behavior may be due to the decreasing the coefficient of friction for all samples that reinforced with nano clay.

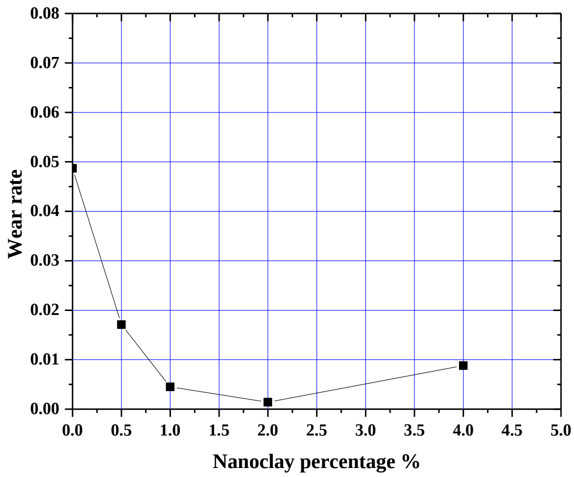


Figure 4 Wear rate test

Scanning Electron Microscopy

The SEM of the fractured surface of the sample with different weight percentages of nanoclay is shown in Fig5. The results showed that the wear resistance of samples reinforced with nanoclay were enhanced due to decreasing the coefficient of friction and the sample reinforced with 2% weight percentage of nanoclay have a higher wear resistance.

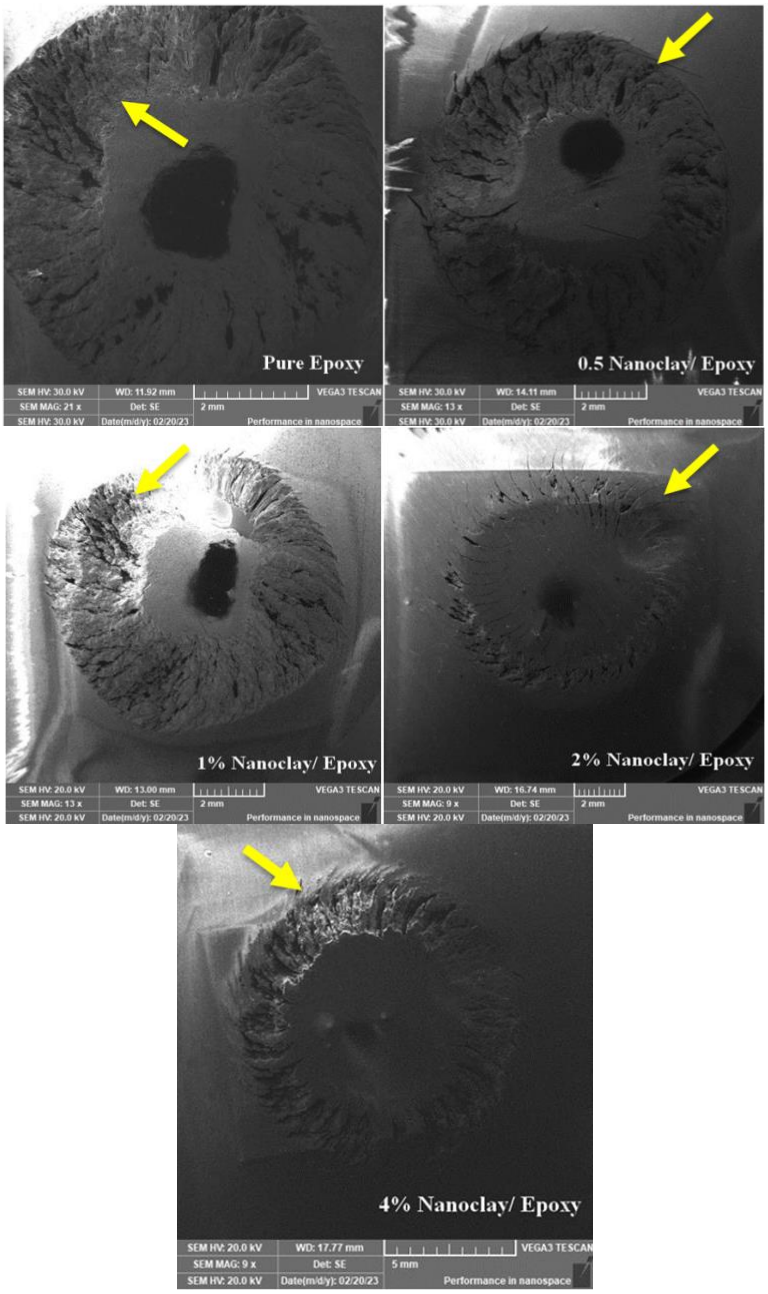


Figure 5 The SEM images of nanocomposite coatings containing

Conclusion

1. In this work, the hardness and tribological properties of the epoxy reinforced with the nanoclay content of up to 4 wt.% were investigated. Both properties increased with increasing content of nanoclays. The improvement in the wear resistance of the nanocoposites coating compared to a pure epoxy can provide an adequate compatibility among novel nanocomposites for surface coatings. A correlation of hardness and friction coefficient with the wear resistance of the nanocomposites with the percentages of intercalated nanoclay.
2. The adhesive tests showed that the adhesive strength decreased with increasing the weight percentage of nanoclay.
3.

References

1. W.-Y. C. D. KUO-CHUNG CHENG, K. C. LIA, "Kinetic Approach for Epoxy Resins Cured with Diaminodiphenyl Sulfone Under Non-isothermal Conditions," J. Appl. Polym. Sci., vol. 71, pp. 721–728, 1998 .

2. C. P. W. S. YANGYANG SUN, ZHUQING ZHANG, KYOUNG-SIK MOON, "Glass Transition and Relaxation Behavior of Epoxy Nanocomposites YANGYANG," J. Polym. Sci. Part B Polym. Phys., vol. 42, pp. 3849–3858, 2004 .

3. A. B. G. Mir Mohammad Alavi Nikjea*, Mohammad Reza Khanmohammadia and and M. Haghshenasb, "Nanosilica reinforced epoxy floor coating composites: preparation and thermophysical characterization," Curr. Chem. Lett., pp. 13–20, 2012 .

4. L. W. Songwei Chen, Bo You, Shuxue Zhou, "Preparation and Characterization of Scratch and Mar Resistant Waterborne Epoxy/Silica Nanocomposite Clearcoat," J. ofAppliedPolymer Sci., vol. 112, pp. 3634–3639, 2009 .

5. S. J. Nathaniel Chisholm, Hassan Mahfuz *, Vijaya K. Rangari, Adnan Ashfaq, "Fabrication and mechanical characterization of carbon/SiC-epoxy nanocomposites," Compos. Struct. 67, vol. 67, pp. 115–124, 2005.

6.Cheng K.C., Lai K.C., and Chiu W. J. J. (1999) Kinetic approach for epoxy resins cured with diaminodiphenyl sulfone under non-isothermal conditions. J. Appl. Polym. Sci., 71, 721-728.

7.May C.A. (1998) Epoxy Resins: Chemistry and Technology. 2nd ed., Marcel Dekker, New York, USA.

8.Lee H., and Nevellie K. (1967) Handbook of Epoxy Resin. McGraw-Hill, New York, USA.

9.Sun Y., Zhang Z., Moon K. S., and Wong C. P. (2004) Glass transition and relaxation behavior of epoxy nanocomposites. J. Polym. Sci. Pol. Phys., 42, 3848-3858.

10.Cheng K.C. (1998) Kinetic model of diepoxides with reactive diluents cured with amines. J. Polym. Sci. Pol. Phys., 36, 2339-2348.

11.Startsev O.V., Krotov A. S., and Startseva L. T. (1998) Interlayer shear strength of polymer composite materials during long term climatic ageing. Polym. Degrad. Stabil., 2, 183-186.

12.Lee J. R., Park S. J., Seo M. K., Baik Y. K., and Lee S. K. (2006) A study on physicochemical properties of epoxy coating system for nuclear power plants. Nucl. Eng. Des., 236, 931-937.

13. Pham, H.Q. and Marks, M.J. (2004) Epoxy resins, in The Encyclopedia of Polymer Science and Technology (on-line addition), John Wiley & Sons, Inc., NewYork, pp. 678-804.

14.Greenlee, S.O. (1948) US Patent 2 456 408.

15. Carpenter, A.S., Colfield, S., Reeder, F., Swansea, K., and Wallsgrove, E.R. (1952)US Patent 2 602 075.

16. Reinking, N.H., Barnabeo, A.E., and Hale, W.F. (1963) J. Appl. Polym. Sci., 7, 2135, 2145, 2153.

17. Brode, G.L. and Wynstra, J. (1966) J. Polym. Sci., Part A-1, 4, 1045.

18. Myers, G.E. and Dagon, J.R. (1964)J. Polym. Sci., Part A, 2, 2631.

19.Hale, W.F. (1969) Phenoxy resins, in Encyclopedia of Polymer Science and Technology, John Wiley & Sons, Inc., New York., pp. 111-122.

20. Poly(hydroxy ether) 1 is manufactured by InChem and is available in various grades sold under the trade names InChem Rez™ Phenoxy Resin PKHH® and PKHM.; see www.phenoxy.com (December, 2008).

21.Salame, M. and Temple, E.J. (1974) Adv. Chem., 135, 2631.

22. Salame, M. (1986) J. Plast. Film Sheeting2,321.

23. Van Hoorn, D.H. (1968) J. Appl. Polym. Sci., 12, 871.

24. Heinemeyer, B.W. and Tatum, S.D. (1986 US Patent 4 612 156.

25. Silvis, H.C., Berman, J., and White, J.E. (1987) US Patent 4 647 648. 26. Silvis, H.C. and Morgan, T.A. (1987) USPatent 4 671 102.

27. Flory, P.J. (1946) Chem. Rev., 39, 137. 19 Shechter, L. and Wynstra, J. (1956) Ind. Eng. Chem., 48, 86.

28. Senthilkumar K, Saba N, Chandrasekar M, Jawaid M, Rajini N, Alothman OY, Siengchin S (2019) Evaluation of mechanical and free vibration properties of the pineapple leaf fibre reinforced polyester composites. Constr Build Mater 195:423–431.

29. Pappu A, Pickering KL, Thakur VK (2019) Manufacturing and characterization of sustainable hybrid composites using sisal and hemp fibres as reinforcement of poly (lactic acid) via injection moulding. Ind Crops Prod 137:260–269

30. Alves C, Silva AJ, Reis LG, Freitas M, Rodrigues LB, Alves DE (2010) Ecodesign of automotive components making use of natural jute fiber composites. J Clean Prod

31. Alves C, Silva AJ, Reis LG, Freitas M, Rodrigues LB, Alves DE (2010) Ecodesign of automotive components making use of natural jute fiber composites. J Clean Prod

32. Abdellaoui H, Bensalah H, Raji M, Rodrigue D, Bouhfid R, El-Kacem QA (2017) Laminated epoxy biocomposites based on clay and jute fibers. J Bionic Eng

33. W.-Y. C. D. KUO-CHUNG CHENG, K. C. LIA, "Kinetic Approach for Epoxy Resins Cured with Diaminodiphenyl Sulfone Under Non-isothermal Conditions," J. Appl. Polym. Sci., vol. 71, pp. 721–728, 1998.

34. F. Pakaya, H. Ardhyananta, and S. T. Wicaksono, ―Mechanical Properties and Thermal Stability of Epoxy/RTV Silicone Rubber,‖ IPTEK J. Technol. Sci., vol. 28, no. 1, p. 7, 2017, doi:
10.12962/j20882033.v28i1.2216.

35. C. P. W. S. YANGYANG SUN, ZHUQING ZHANG, KYOUNG-SIK MOON, “Glass Transition and Relaxation Behavior of Epoxy Nanocomposites YANGYANG,” J. Polym. Sci. Part B Polym. Phys., vol. 42, pp. 3849–3858, 2004.

36. A. B. G. Mir Mohammad Alavi Nikjea*, Mohammad Reza Khanmohammadia and and M. Haghshenasb, “Nanosilica reinforced epoxy floor coating composites: preparation and thermophysical characterization,” Curr. Chem. Lett., pp. 13–20, 2012.

37. L. W. Songwei Chen, Bo You, Shuxue Zhou, “Preparation and Characterization of Scratch and Mar Resistant Waterborne Epoxy/Silica Nanocomposite Clearcoat,” J. ofAppliedPolymer Sci., vol. 112, pp. 3634–3639, 2009.

38. S. J. Nathaniel Chisholm, Hassan Mahfuz *, Vijaya K. Rangari, Adnan Ashfaq, “Fabrication and mechanical characterization of carbon/SiC-epoxy nanocomposites,” Compos. Struct. 67, vol. 67, pp. 115–124, 2005.

39. S. M. Hashim and W. B. Salih, ―Study of effect reinforcement by coconut fiber on some mechanical and physical properties of thermoset polymer,‖ IOP Conf. Ser. Mater. Sci. Eng., vol. 757, no. 1, 2020, doi1757899/10.1088 :X/757/1/012020 .

40. Annual Book of ASTM Standard, ―Rubber Property—Durometer HardnesMaterials, E. I., Manufacturing, C. B., Hardness, D & ,.Laboratories, C. (2017). Rubber Property—Durometer Hardness1 Methods, S. T. (2008). Standard Test Methods for Rubber Property-Compression Set 1, i(Reapproved), 1–6.,‖ Astm D 2240, pp. 1–13 ,2015, doi: 10.1520/D2240-15.2.

41. Annual Book of ASTM Standard, ―D4541-09: Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion,‖ ASTM Int., pp. 1–16, 2009, doi: 10.1520/D4541-09E01.2

42. Annual Book of ASTM Standard, ―ASTM D638: Standard Test Method for Tensile Properties of Plastics,‖ ASTM Stand., no. January ,pp. 1–15, 2003.