Evaluation of Composites from Dimethylol Urea/Hydroxylated Vegetable Oils for Possible Application for Water Resistant Emulsion Paint.

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INTRODUCTION

The development of water-resistant emulsion paints has gained significant attention in recent years due to the growing demand for sustainable and environmentally friendly coatings (Lee, 2021). Dimethylol urea (DMU) is a promising binder material due to its excellent adhesion properties and resistance to water and chemicals (Osemeahon & Barminas, 2022). However, its brittleness and formaldehyde emission limit its application (Achi, 2003). Hydroxylated vegetable oils (HVOs), on the other hand, offer excellent flexibility and water resistance (Manawwer, 2024). By combining DMU and HVOs, it is possible to create composites with improved properties.

The composites of DMU and HVOs have been shown to exhibit improved water resistance and adhesion properties compared to the individual components (Shahla, 2021). The reaction between DMU and HVOs leads to the formation of a new polymer network, which enhances the thermal stability and flexibility of the composites (Gurses, 2024). These improved properties make the DMU/HVO composites an attractive alternative to traditional binders for water-resistant emulsion paint (Udeozo, 2023).

The use of vegetable oils in paint formulations has been widely reported (Surajudeen and Zebulu, 2025). Vegetable oils offer excellent flexibility and water resistance, making them an ideal component for water-resistant emulsion paint (Manawwer, 2024). The hydroxylation of vegetable oils enhances their reactivity and compatibility with other polymers, making them suitable for use in composites (Shahla, 2021).

The use of paint on surfaces has gained global attention (Osemeahon & Barminas, 2022). The terms “paint” and “surface coating” are often used interchangeably. However, surface coating is the more general description of any material that may be applied as a thin continuous layer to a surface. Study has shown that Paint was traditionally used to describe pigmented materials as distinct from clear films which are more properly called lacquers or varnishes.

Paint is a loose word covering a whole variety of materials; enamels, lacquers, varnishes, undercoats, surfaces, primers, sealers, fillers, stoppers and many others. If the pigment is omitted, the material is usually called a varnish. The pigmented varnish – the paint – is sometimes called an enamel, lacquer, finish or topcoat, meaning that it is the last coat to be applied and the one seen when the coated object is examined. (Surajudeen & Zebulu, 2025).

Paints are complex mixtures of chemicals that serve both decorative and protective purposes, enhancing the aesthetic value and durability of surfaces (Ashok Kumar, 2013). Paints are of different categories, such as oil-based and water-based paints, and their applications in various industries, including construction, automotive, and industrial coatings (Shahla, 2021). Paint can therefore be defined as a solution or suspension (emulsion) of pigment, binder, and mineral solvent (or water) that on drying forms an adhering film on the surface. It is applied for protection and/or decoration.

The development of sustainable and environmentally friendly coatings is a growing area of research (Yousefi 2021). The use of DMU/HVO composites in water-resistant emulsion paint offers a promising solution to the

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environmental concerns associated with traditional coatings (Lee, 2021). It is against this background that the current study focuses on Evaluation of Composites from Dimethylol Urea/Hydroxylated Vegetable Oils for Possible Application for Water Resistant Emulsion Paint.

Statement Of the Problem

The pressing issue of climate change and environmental pollution from industrial chemicals and products demands immediate attention. Volatile organic compounds (VOCs) are significant contributors to this problem, with coating surfaces accounting for 30% of environmental VOCs (Osemeahon & Barminas, 2022). While the development of water-resistant emulsion paints has gained significant attention in recent years due to the growing demand for sustainable and environmentally friendly coatings (Lee, 2021); it is important to 0identify processes that can scientifically address problems associated with the development of the said water-resistant emulsion paints.

Evidence from many scientific studies revealed that, while coatings and paints have numerous benefits, solvent-based paints pose environmental and health risks due to Volatile organic compounds (VOCs) emissions during the drying process (Barminas, & Osemeahon, 2023). The identified environmental and health risks due to Volatile organic compounds (VOCs) are said to constitute numerous environmental problems (Achi, 2023).

In contrast, water-based paints are more environmentally friendly but often compromise on performance and durability which necessitates study of this magnitude more especially, the Evaluation of Composites from Dimethylol Urea/Hydroxylated Vegetable Oils for Possible Application for Water Resistant Emulsion Paint.

Given the trade-offs between quality and safety, there is a pressing need to develop a novel emulsion paint binder that combines the benefits of solvent-based paints with the safety and environmental advantages of water-based paints. It is against these problems that research hope to address this challenge by exploring the potential of hydroxylated vegetable oils and dimethylol urea copolymers as a sustainable binder for emulsion paints.

Aim And Objectives

The aim of this study is to Evaluate Composites from Dimethylol Urea/Hydroxylated Vegetable Oils for Possible Application for Water Resistant Emulsion Paint. Specifically, the study seeks to:

- **i.** Extract coconut oil, black seed oil, mahogany oil, and sorrel oil from their respective seeds.
- **ii.** Synthesize dimethylol urea.
- **iii.** Epoxidize and hydroxylate the extracted vegetable oils.
- **iv.** Copolymerize hydroxylated vegetable oils with synthesized dimethylol urea resin.
- **v.** Characterize the resulting copolymer.
- **vi.** Formulate emulsion paint using the copolymer resin as a binder.
- **vii.** Evaluate the physical properties of the formulated emulsion paint and compare them with standard values.

LITERATURE REVIEW

Paint

Paint is any liquid, liquefiable or mastic composition which after application to a substrate in a thin layer is converted to an opaque solid film. Painting has been known to be one of the most important aspects of building

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construction. It covers the block work and concrete rendering (plaster) with attractive and beautiful colours, giving the building a high aesthetic value that makes it decorative, clean and habitable. Paint is indispensable in building construction all over the World (Opara, 2024).

The terms "paint" and "surface coating" are often used interchangeably. Surface coating is the more general description of any material that may be applied as a thin continuous layer to a surface. Paint was traditionally used to describe pigmented materials as distinct from clear films which are more properly called lacquers or varnishes. Paint is a loose word covering a whole variety of materials; enamels, lacquers, varnishes, undercoats, surfaces, primers, sealers, fillers, stoppers and many others. If the pigment is omitted, the material is usually called a varnish. The pigmented varnish – the paint – is sometimes called an enamel, lacquer, finish or topcoat, meaning that it is the last coat to be applied and the one seen when the coated object is examined. (Surajudeen and Zebulu, 2025)

Paint can also be defined as a solution or suspension (emulsion) of pigment, binder, and mineral solvent (or water) that on drying forms an adhering film on the surface. It is applied for protection and/or decoration. Paints and coatings can be classified into the following major categories such as Architectural coatings, Product coatings for original equipment manufacturers and Special purpose coatings. Of these three major categories, emulsion paints fall in the first category.

Constituents of paint

Paint is made up of three major constituents:

- **i.** Pigment: This gives paint its colour and opacity.
- **ii.** Binder: This holds the pigment and other additives together.
- **iii.** Solvent: the solvent gives the paint its ability to be spread over a surface.
- **iv.** Additives: these are other ingredients added to paint to provide a specific purpose (Igwebike, 2022).

Pigment: the purpose of the pigment, from a decorative standpoint, is to obliterate the surface over which the product is applied and provide colour to it. From a protective standpoint for exterior use, serve the function of protecting the vehicle from degradation by ultraviolet radiation. Most clear films of drying oil, resin or oleoresinous compositions are not likely to withstand sunlight exposure for more than twelve months. Pigment films, however, will protect for a considerable number of years. Certain pigments are used for their unique chemical functions such as rust inhibition on meta structures and control of fouling on ship bottoms. Pigments are also classified according to the type or colour. Early classifications distinguished between mineral or natural pigments which came from the earth and were either ground directly or refined to a certain extent; and chemical pigments which required a conversion of one sort or another.

The main functions of pigments are to impart colour and hiding, provide opacity or covering power to the paint, improve hardness and durability, and to aid the film-former or vehicle (non-vehicle) in protecting the surface.

Binder: The binder is the film-forming component of paint (Vermeer's Palette, 2015). These are materials used in paint production that are capable of bonding together all the particles of pigment that are either coated or suspended in a liquid when the paint is exposed to air. Binders impart adhesion, bind the pigments together and strongly influence such properties as gloss potential, exterior durability and toughness. The most common binders in paint manufacture remain Styrene Acrylics. They come either as an emulsion or as a copolymer. Styrene acrylics have good film forming and binding properties with excellent resistance towards alkali and water. Styrene acrylics find their applications in both interior and exterior paints with good washability. Styrene acrylics are also used in the manufacture of floor tiles, building adhesives, roof-coatings and some sealants.

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The binder is the only component that is always present among all the various types of formulations. Many binders are too thick to be applied and must be thinned. The type of thinner if present varies with the binder. The binder imparts such properties as gloss, durability, flexibility, and toughness.

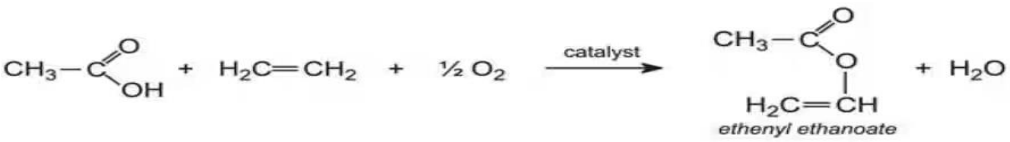
Binders include synthetic or natural resins such as alkyds, acrylics, vinyl-acrylics, vinyl-acetate/ethylene (VAE), polyurethanes, polyesters, melamine resins, epoxy, silanes or siloxanes or oils. Binders can be categorized according to the mechanisms for film formation. Thermoplastic mechanisms include drying and coalescence. Drying refers to simple evaporation of the solvent or thinner to leave a coherent film behind. Coalescence refers to a mechanism that involves drying followed by actual interpretation and fusion of formerly discrete particles. Thermoplastic film-forming mechanisms are sometimes described as “thermoplastic cure” but that is a misnomer because no chemical curing reactions are required to knit the Systems." Thermosetting mechanisms, on the other hand, are true curing mechanisms that involve chemical reaction(s) among the polymers that make up the binder (Babajide, Bodunde, & Salami, 2024).

The three most important binders (resins) used in modern paints are: acrylic polymers (resins), alkyd polymers (resins) and epoxy polymers (resins)

Acrylic Polymers (Resins)

The binder in many emulsion paints is based on homopolymers or co-polymers of ethenyl ethanoate (vinyl acetate) and a propenoate (acrylic) ester.

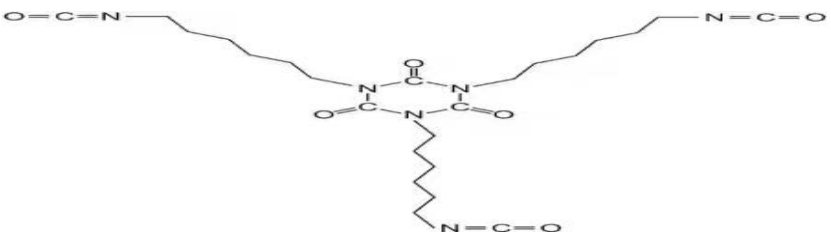
Ethenyl ethanoate is manufactured by passing a mixture of ethanoic acid vapour, ethene and oxygen over heated palladium(ll) and copper(ll) chlorides:



Ethenyl ethanoate and an acrylic ester (for example, methyl 2-methylpropenoate) is then co-polymerized to form a random array, in which these groups link into a linear chain:

Other acrylic esters used as co-monomers with ethenyl ethanoate are ethyl propenoate, butyl propenoates, or a co-polymer of butyl propenoate and methyl 2-methylpropenoate.

Acrylic resins may also be used in industrial paints, either as water-borne emulsion paints or as solvent-borne paints. Solvent-borne industrial paints can have a tough protective finish and are widely used in industry as topcoats, for example on car bodies. The paint frequently comes as two components which are mixed together just before use: the main paint portion typically consists of an acrylic resin produced by the polymerization of a propenoate ester formed from a polyhydric alcohol (diols and triols). The resulting polyester has numerous hydroxyl groups (-OH) pendant from the polymer backbone. The hydroxyl groups react with the other compound often consisting of a polymeric isocyanate such as a trimer of 1,6-diisocyanatohexane (hexamethylene diisocyanate):



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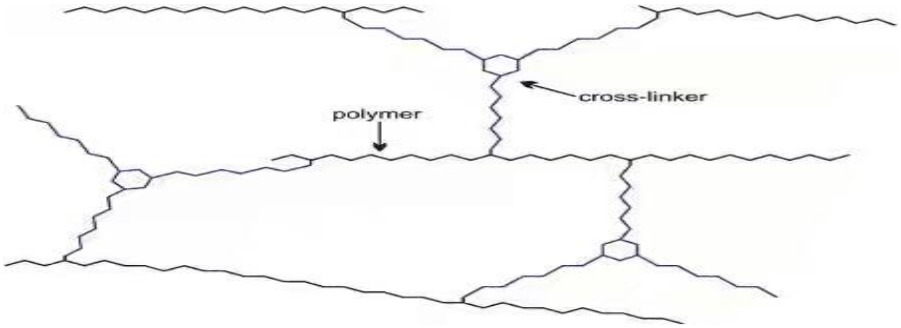
Such a compound is known as a crosslinker for it produces, on reaction with the resin, a three-dimensional structure similar to the polyurethane formed from a polyol and an isocyanate. When these two components are mixed together, a chemical reaction takes place between the hydroxyl groups on the polymer (acrylic resin) and the isocyanate groups on the cross linker:

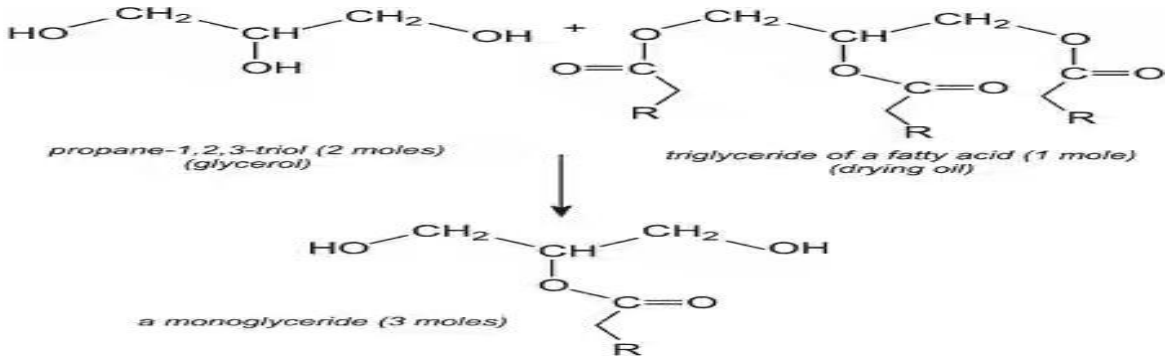


This reaction proceeds relatively slowly at room temperature, allowing enough time for the paint to be applied, after which the solvent thinner evaporates, and the painted item is placed in an oven to accelerate the chemical reaction. This greatly increases the molecular mass of the polymer causing it to become a three-dimensional molecule and form a hard film, resistant to chemicals.

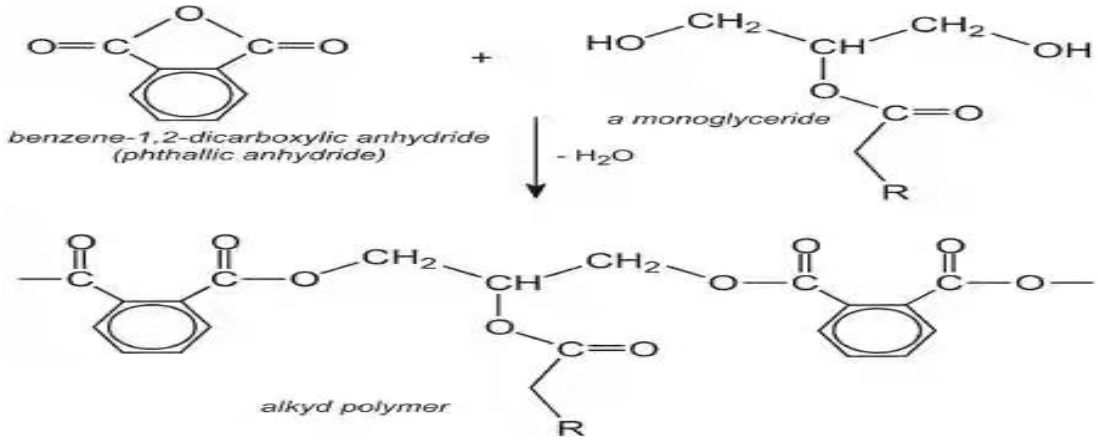
Alkyd Polymers (Resins)

Decorative gloss paints typically contain alkyd polymers (resins). A typical resin is produced from a polyol such as propane-1,2,3-triol (glycerol) with a dibasic acid such as benzene-1,2-dicarboxylic (phthalic) anhydride and a drying oil (linseed or soybean oil). On being heated together, ester linkages are formed, and water is a by-product. The name alkyd is derived from alcohol and anhydride.

The first step in making the alkyd polymer is the reaction between the triol and the drying oil to produce a monoglyceride. For example:



The monoglyceride then reacts with the anhydride to form the alkyd polymer (resin):



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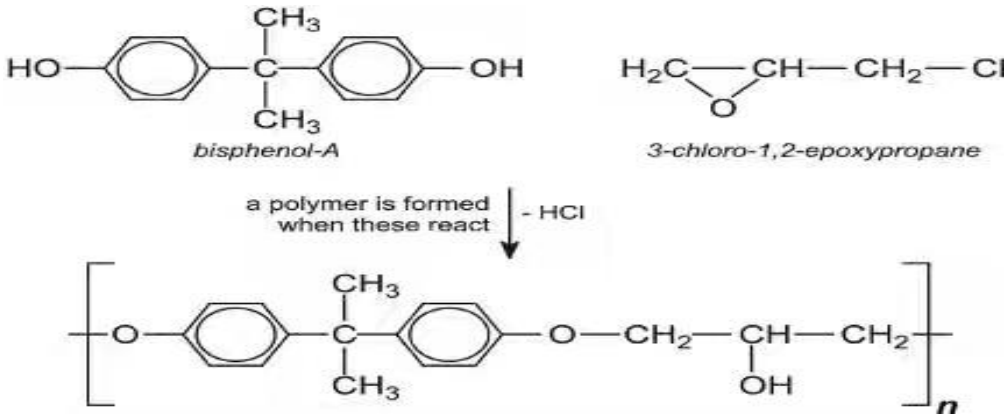
The alkyd resins, which generally have relative molecular masses in the range of 10 000 - 50 000, are usually carried in organic solvents (solvent-borne paints). Turpentine extracted from trees was used in the past as the solvent, but this has been replaced by solvents from petrochemical feedstock, such as 'white spirit' which is a mixture of aliphatic and alicyclic hydrocarbons.

Once the alkyd resin is applied, the pendant oil drying groups react with oxygen in the air to form a cross-linked, hard thermoset coating, with a high molecular mass.

Epoxy polymers (resins)

Epoxy resins are often used as the binder in industrial coatings (primers). They give the paint excellent adhesion together with high resistance to chemicals (corrosion), and physical resistance necessary, for example, on ships and chemical storage tanks.

The epoxy resins are made from 1-chloro-2,3-epoxypropane (produced from 3-chloropropene) and substituted phenols, such as bisphenol A:



The value of n can be controlled to give a range of resins varying from viscous liquids to solids with high melting points. Epoxy resins can be carried in solvents such as aromatic hydrocarbons, alcohols, ketones and esters (solvent-borne paints) or as dispersions in water (water-borne paints) as true emulsions. They are not normally used in topcoats for outdoors because they are susceptible to UV degradation, but they make excellent interior coatings and exterior primers. Epoxy resins are also used as adhesives (e.g. Araldite) and electrical insulators.

In the constituents for production of emulsion paints, binder (PVA) usually takes between 17 to 33% total cost of production depending on paint type and this raw material is not available locally, therefore imported. Hence, high cost of this raw material (binder) and consequently high cost of production due to the use of expensive binder. In addition, PVA is not environmentally friendly because of its non-biodegradability which can contaminate the environment if not properly handled. This is because PVA releases toxic fumes when burned. It can be hazardous to the environment, especially if it is mixed with water and comes in contact with fish. Polyvinyl acetate, poly (ethenyl ethanoate), poly (1-acetyloxiethylene) is a rubbery synthetic polymer with the formula $(C_4H_6O_2)_n$. It has a molar mass of 86.09g/mol/unit. It belongs to the polyvinyl esters family with the general formula: $RCOOCHCH_2$. It is a type of thermoplastic used as an emulsion in water, and as a binder in paints and coatings. PVA emulsions are used as adhesives for porous materials, particularly for wood, paper, and cloth, and as a consolidant for porous building stone, in particular sandstone (Surajudeen & Zebulu, 2025).

Vehicle: there are two vehicles. The volatile and non-volatile vehicle.

Non-volatile vehicle: this constituent of paint in most cases is composed of drying oils, resins or a combination of the two as used in latex glossy paint. A few types of paints have inorganic vehicles. It is often referred to as the binder or medium and is the material responsible for film formation as the paint dries, that is, becomes covered from a liquid or semi-liquid layer on the substrate to a hard coherent solid film.

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The purpose of the non-volatile vehicle is to impart cohesion and adhesion to the paint film, convert from the liquid coating to a solid dry film, act as a moisture barrier thereby providing resistance to water, chemicals and abrasion, provide gloss to the coatings and to bind the pigment particles together.

Volatile vehicle: This is also known as solvent. It usually consists of hydrocarbon solvents or water; it is used to lower the viscosity of the composition for ease of application. Organic solvents other than hydrocarbons, including ketones and esters are necessary in some types of composition (Udeozo, Umedum, Okoye, & Kelle, 2023).

According to Surajudeen and Zebulu, (2025), paint could be applied as a solid, a gaseous suspension (aerosol) or a liquid. Techniques vary depending on the practical or artistic results desired. As a solid (usually used in industrial and automotive applications), the paint is applied as a very fine powder and then baked at high temperature. This melts the powder and causes it to adhere (stick) to the surface. The reasons for doing this involve the chemistry of the paint, the surface itself, and perhaps even the chemistry of the substrate (the overall object being painted). This is commonly referred to as "powder coating"-an object. As a gas or gaseous suspension, the paint is suspended in solid or liquid form in a gas that is sprayed on an object. The paint sticks to the object. This is commonly referred to as "spray painting" an object. The reasons for doing this include: The application mechanism is air and thus no solid object ever touches the object being painted, The distribution of the paint is very uniform so there are no sharp lines, It is possible to deliver very small amounts of paint, Chemical (typically a solvent) can be sprayed along with the paint to dissolve together both the delivered paint and the chemicals on the surface of the object being painted and Some chemical reactions in paint involve the orientation of the paint molecules.

In the liquid application, paint can be applied by direct application using brushes, paint rollers, blades, other instruments, or body parts such as fingers. Paint application by spraying is the most popular method in the industry. In this, paint is atomized by the force of compressed air or by the action of high-pressure compression of the paint itself, which results in the paint being turned into small droplets which travel to the article which is to be painted. Manually, rollers are used to apply paints to surfaces. Rollers generally have handles that allow for different lengths of poles which can be attached to allow for painting at different heights. Generally, roller applications take two coats for even colour. A roller with a thicker nap is used to apply paint on uneven surfaces. Edges are often finished with an angled brush (Opara, 2024).

Types of paint

Basically, there are two categories of paint:

1. Oil paint which is oil based
2. Emulsion or water-based paint.

Paints or coatings can further be classified according to their end use (decorative, auto, industrial and marine), in terms of solvent used (water based and solvent based paint), and higher performance or special type of paint (insecticidal based, antimicrobial based, fireproof-based paint). The quality of paint or coating is judged by the length of time or duration during which the paint maintains its decorative and protective value (Adetuyi, Ajekwene, & Jabar, 2022).

Oil-based paint

Oil paint is a type of slow-drying paint that consists of particles of pigment suspended in a drying oil, commonly linseed oil. The viscosity of the paint may be modified by the addition of a solvent such as turpentine or white spirit, and varnish may be added to increase the glossiness of the dried oil paint film. Oil paints have been used in Europe since the 12th century for simple decoration but were not widely adopted as an artistic medium until the 15th century. Common modern applications of oil paint are in finishing and protection of wood in buildings and exposed metal structures such as ships and bridges. Its hard-wearing properties and luminous colours make it desirable for both interior and exterior use on wood and metal. Due to its slow-

drying properties, it has recently been used in paint-on-glass animation. Thickness of coat has considerable bearing on time required for drying: thin coats of oil paint dry relatively quickly. The technical history of the introduction and development of oil paint, and the date of introduction of various additives (driers, thinners) is still-despite intense research since the mid18th century-not well understood (Afzal, Siddiqi, Saeed, & Ahmad, 2023).

Until 1991, nothing was known about the organic aspect of cave paintings from the Paleolithic era. Many assumptions were made about the chemistry of the binders. The oldest known oil paintings date from 650 AD, found in 2008 in caves in Afghanistan's Bamiyan Valley. Using walnut and poppy seed oils (Adetuyi, Ajekwene, & Jabar, 2022).

Emulsion or water-based paint

The current global paint and coating industry is doing its best to build a safer, greener and more sustainable world. Abdullah, & Park, (2023) noted that industrial activity has a significant impact on economic and social development and the environment around the world. Also, the global paint industry has been making the shift to more environmentally advanced technologies for more than fifty years. He also reported that today, clean water-based paint technologies are used around the world accounting for 70% of the total US paints and coating market while 30% are growing across Asia (Opara, 2024).

Among the basic methods that have been used in the production and application of coatings with volatile organic compounds omission (VOCs), replacement of volatile organic compounds (VOCs) with water in emulsion paints is the cheapest and most sustainable procedure compared to powder and UV-drying coating (Hutanu, Woods & Darie, 2023). Water paints, though fundamentally inferior in their properties compared to oil paint, is environmentally friendly (Kumthekar, & Kolekar, 2021).

In recent decades, conventional paints are increasingly replaced by environmentally friendly formulas, whose use is recommended due to ecological considerations, specifically the reduction of VOCs and economic aspects (low volumes of organic solvents which tend to have limited availability and are expensive). These types of paints include powder, high-solids, polyurethanes, radiation curable, emulsion paints etc. owing to their reduced solvent content, the water-soluble paints are characterized by low VOCs emission, non-inflammability. Even though they could possess a higher viscosity and require more time and heat to dry, the quality of the resulting films are comparable and sometimes superior to those obtained with solvent soluble paints. The first coatings with water-based paints were the emulsion paints containing different resins. They showed the disadvantages of not being suitable for metal (due to the small amounts of water remaining in film and on the support which caused corrosion phenomena) and having a low mechanical stability (Andrei, Irina, Cristina-Gabriela, Lidia, Teodorescu, & Lucian 2025).

Emulsion house paint is a water-based paint principally used for internal and external surface coatings, mostly in buildings for appearance and protection. The processes involved in paint production, quality and performance of emulsion paint depend largely on the properties of its constituents and the ratios of these constitutions. The constituents generally used for the production of emulsion house paints include; prime pigments, solvents, extenders pigments, binders and additives (Surajudeen and Zebulu, 2025).

Emulsion paint is paint in which water is used in place of organic solvents as thinner. It is an emulsion of two phases, one of which is water and this paint can easily be diluted or thinned with water, because water is the continuous phase present in it. The vehicle or surface forming material (a synthetic resin latex) is dispersed in water by means of a dispersing agent acting as a binder. It may also have in it another vehicle or film forming constituent such as oil. The binder may thus be oil, oleoresinous varnish or other emulsifiable binders. Pigment and extenders are dispersed in such an emulsion. In addition to these constituents, emulsion paint may also have emulsifying agents or surface-active agents, stabilizers, driers, antifoaming agent, preservatives etc (Sharma, 2021).

Emulsion paints offer the easiest application and soap-and-water cleanup, are the fastest drying and have less odour than solvent paints. Latex performs well on interior walls and ceilings; its quick drying time prevents the

kind of ripples and sags that can occur with slower-drying alkyd finishes. When a thin film of emulsion paint is applied to a surface, water undergoes evaporation from the surface breaking of emulsion takes place. The resin particles coalesce to form a continuous homogenous clear film. Emulsion paint consists of an emulsion of two phases, one which is water.

In such paints, continuous phase consists of water. Thus, the paint can be easily diluted or thinned with water. The drying of emulsion film occurs in two stages. In the first stage, the film remains permeable to water, and moisture escapes, while in the second stage, drying of resin takes place by oxidation. In order to stabilize the emulsion, surfactants are added as emulsifiers. The stability of the paint is improved by the addition of high molecular weight materials such as gum Arabic, starch, water soluble cellulose derivatives and proteins. These act as protective colloids. Synthetic resins or polymeric materials like latex, polyvinyl acetate and acrylic esters are also added to give strength to the films. On application, water evaporates leaving a thin film of the pigment, resin and oil. Emulsion paints have better hiding power, need no other thinner than water, are easy and quick drying. These paints can be very easily applied with brush or roller and after painting; the brush and roller can be easily washed with water. The surface, over which emulsion paint is applied, can be easily washed with water (Sharma, 2021).

Emulsion or water-based latex paint is usually used to paint interior walls and ceilings. Some types of emulsion paint can also be used to paint woodwork. They are used for special finishes, such as rag rolling. It can be used for stenciling and sponge painting too. Emulsion paint usually has at least a slight sheen to it. This paint type comes in several different finishes, including high gloss, matte, eggshell, silk and satin. Typically, the higher the gloss of the paint the easier it is to clean, which is why many people use gloss or satin finishes on doors and windows.
Because it is water-based, emulsion paint has less fumes and is easier to clean up than oil-based paint varieties. Running brushes under hot water cleans off all the paint. It also dries much more quickly than oil-based paint unless in a humid environment.

One of the disadvantages of emulsion paint is that it is usually more expensive than oil-based varieties. However, many people are willing to pay more for water-based paint to avoid dealing with oil-based paint's heavy fumes and the need to clean the brushes with turpentine. Water-based paint includes some vinyl and acrylic resins to make it more durable.
Waterborne paints, which are widely used for protection and decoration, have quite a number of advantages. These include easy application, quick drying qualities, lack of undesirable odour, good washability and excellent finish. These paints also eliminate the use of organic solvents for thinning. They also have the advantage of utilizing a wide range of natural and synthetic resins at high concentrations (Ansari, & Goswami, 2023).

Volatile organic compounds (VOCs)

VOCs may be natural or synthetic. Like organic chemicals in general, there are many different compounds which may be classified as VOCs. The compounds the nose detects as smells are generally VOCs. Modern industrial chemicals such as fuels, solvents, coatings, feedstock, and refrigerants are usually VOCs.

There is no clear and widely supported definition of a VOC. From a chemistry viewpoint "Volatile Organic Compound" can mean any organic compound (all chemical compounds containing carbon) that is volatile (evaporating or vaporizing readily under normal conditions). This is a very broad set of chemicals. Definitions vary depending on the context. There are many other widely used terms that are a subclass of VOCs. Laws or regulations are often responsible for creation of legal definitions of VOCs or definitions of subclasses of VOCs.

Health Canada classifies VOCs as organic compounds that have boiling points roughly in the range of 50 – 250^{O}C (122 to 482^{O}F). The emphasis is placed on commonly encountered VOCs which would have an effect on air quality.

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According to European Union, a VOC is any organic compound having an initial boiling point less than or equal to 250^{O}C measured at a standard atmospheric pressure of 101.3kpa and can do damage to visual or audible senses.

United States – VOCs (or specific subsets of the VOCs) are legally defined in the various laws and codes under which they are regulated.

In most countries a separate definition of VOCs is used with regard to indoor air quality that comprises each organic chemical compound that can be measured as follows:

- Adsorption from air on Tenax TA.

- Thermal desorption.

-Gas chromatographic separation over a 100% non-polar column (dimethylpolysiloxane). VOCs are all compounds that appear in the gas chromatogram between and including n – hexadecane. Compounds appearing earlier are called VVOC (very volatile organic compounds). Compounds appearing later are called SVOC (semi – volatile organic compounds) (AQT, 2015 – 2017).

Sources of VOCs

Volatile organic compounds are produced naturally through biological mechanisms such as metabolism. Industrial use of fossil fuels produces VOCs either directly as products (e.g. gasoline) or indirectly as byproducts (e.g. automobile exhaust). It has also been proven that trees release Volatile Organic Compounds (VOCs). That VOC molecules form tiny crystals or aerosols in the forest atmosphere, and that in the absence of other aerosols, these crystals serve as Cloud Condensation Nuclei (CCN), attracting a great deal of water vapor and forming large, heavy droplets that rapidly precipitate in the same region in which they formed in a fast and efficient manner. The importance of these natural VOC emissions that form CCNs lies in the hydrologic cycle: it is estimated that at least 20 to 30 percent of all the rainfall in the region results from this process (Joao, 2022).

Some examples of VOC sources are as follows:

- **Methane:** The most common VOC is methane, a greenhouse gas sometimes excluded from analysis of other VOCs using the term non – methane VOCs, or NMVOCs. Major worldwide sources of atmospheric methane include wetlands ruminants such cows, energy use, rice agriculture, landfills, and burning biomass such as wood. Methane is the primary component of natural gas (AQT, 2015 – 2017).

- **Formaldehyde:** Since people today spend most of their time at home or in an office, long – term exposure to VOCs in the indoor environment can contribute to sick building syndrome. Many building materials such as paints, adhesives, wall boards, and ceiling tiles emit formaldehyde, which irritates the mucous membranes and can make a person irritated and uncomfortable. There are also many sources of VOCs in office buildings, which include new furnishings, wall coverings, and office equipment such as photocopy machines which can off – gas VOCs into the air (AQT, 2015 – 2017).

- **Vegetation:** Plants in physiological process emit Volatile Organic Compounds (VOCs) known as terpenes into the atmosphere. VOC emissions from vegetation are estimated to be comparable or exceed those from anthropogenic aerosols at a regional and global level. Volatile organic compounds include saturated and unsaturated non – metallic hydrocarbons and oxygenated hydrocarbons, such as carboxylic acids, aldehydes, ketones, ethers, esters, and alcohols. A large number of oxygenated compounds have been found in emissions from plants. Inventories for plant emissions show that isoprene and monoterpenes, classified as isoprenoids, are among the most abundant compounds, followed by alcohols and carbonyls. VOCs such as monoterpenes, esquiterpenes, alcohols, acids, aldehydes, ketones,and esters are stored in different plant organs. Plants, which during photosynthesis fix from 0.5 to 2 percent of carbon, emit high levels of VOCs, leading to a loss of up to 20 percent of the fixed carbon.

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Emissions of dimethyl sulfide (DMS) from the ocean and of non – methane hydrocarbons (NMHCs) from terrestrial vegetation are the main biogenic aerosol sources, followed by their oxidation in the atmosphere. The amount and composition of terpenes and other biogenic hydrocarbons depend on climatic parameters such as temperature and solar radiation and may change radically as a result of changes in the type of vegetative cover due to land use or climate change. The production of aerosol precursor gases depends upon the oxidants in the atmosphere, and their removal is influenced by the dynamics of clouds and precipitation (Joao, 2012).

Health risks of VOCs

Releases, intentional or not, of VOCs may affect the environment or human health, depending on the particular chemicals involved, the quantities and concentrations, and the relative locations of "receptors" which are sensitive to the particular chemical (AQT, 2015 – 2017).

The emissions of VOCs from biomass burning have a significant impact on the health of the population living near the sources or in the downwind regions (Kumud and Sahu, 2014).

Some examples of VOCs causing health or environmental effects are as follows:

i. Atmosphere

ii. Ground water

iii. Indoor air quality

VOC Sensors, principles and measurements

VOCs in the environment or certain atmospheres can be detected based on different principles and interactions between the organic compounds and the sensor components. There are electronic devices that can detect ppm concentrations despite the non – selectivity. Others can predict with reasonable accuracy the molecular structure of the volatile organic compounds in the environment or enclosed atmosphere (Martinez-Hurtado, Davidson, Blyth, & Lowe, 2023).

Direct injection mass spectrometry techniques are frequently utilized for the rapid detection and accurate quantification of VOCs (Biasoli, Yeretzian, Mark, Dewulf, & Van, 2021).

PTR – MS is among the methods that have been used most extensively for the online analysis of biogenic and antropogenic VOCs (Ellis, & Mayhew, 2024). Recent PTR – MS instruments based on time – of – flight mass spectrometry has been reported to reach detection limits of 20pptv after 100ms and 750ppqv after 1min measurement (signal integration) time. The mass resolution of these devices is between 7000 and 10,500m/Δm, thus it is possible to separate most common isobaric VOCs and quantify them independently (Biasoli, Yeretzian, Mark, Dewulf, & Van, 2021).

To achieve comparability of VOC measurements, reference standards traceable to SI-units are required. For a number of VOCs, gaseous reference standards are available from specialty gas suppliers or national metrology institutes, either in the form of cylinders or dynamic generation methods. However, for many VOCs, such as oxygenated VOCs, monoterpenes, or formaldehyde, no standards are available at the appropriate amount of fraction due to the chemical reactivity or adsorption of these molecules. Currently, several national metrology institutes are working on the lacking standard gas mixtures at trace level concentration, minimizing adsorption processes, and improving zero gas. The final scopes are for the traceability and the long-term stability of the standard gases to be in accordance with the data quality objectives (DQO, maximum uncertainty of 20% in this case) required by the WMO/GAW program (Ellis, & Mayhew, 2024).

General uses of VOCs

Volatile organic compounds are generally useful as: Fuels, Solvents, Scents, Precursors, Propellants. Refrigerants, Drugs, Pesticides and Markers

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The wide range of VOCs allows organic materials or processes to be identified by combinations of VOCs they may contain (or especially emit to the atmosphere).

Urea formaldehyde

Urea formaldehyde is a non-transparent thermosetting resin or plastic. It is produced from urea and formaldehyde. These resins are used in adhesives, finishes, particle board, MDF, and molded objects. Urea formaldehyde and related amino resins are a class of thermosetting resins which urea formaldehyde make up 80% produced globally. Examples of amino resins use include in automobile tires to improve the bonding of rubber to tire cord, in paper for improving tear strength, in molding electrical devices, jar caps, etc (Deim, *et al*., 2012).

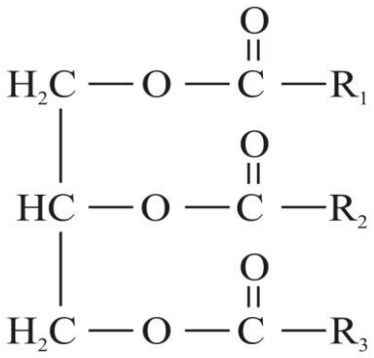
Urea formaldehyde (UF) is a cheap water soluble resin, which cures to form a clear, glossy and hard thermosetting film. It is suitable as a binder for water paint, but intrinsic properties such as formaldehyde emission, brittleness and moisture uptake limits its application (Suurpere, *et al*., 2006). Durability problems associated with UF films is due to its brittleness and poor water resistance, while formaldehyde emission above some threshold concentration is hazardous (Salthammer, *et al*., 2010).

Since urea formaldehyde is soluble in water, its full utilization is needed to meet different requirements of the coating industry in terms of emulsion paint formulation and consequent reduction in VOCs in the environment. This can be achieved if the inherent problems of brittleness, poor water resistance and formaldehyde emission associated with urea formaldehyde resin are addressed (Osemeahon and Dimas, 2014).

Vegetable Oils

The industry has come under increasing pressure to make chemical production more eco-friendly and efforts to shift the prime resource base of industry from fossil (non-renewable) to renewable feed stocks have recently gained momentum due to the rapid rise in the costs of mineral oil and the increasing concerns about the depletion of these resources in the near future. Polysaccharides (cellulose, hemicelluloses and starch), sugars, proteins, wood and plant oils are just a few examples of renewable raw materials to which academic and industrial researches have been devoting increasing attention. Amongst them, vegetable oils are the most widely used for the chemical and polymer industries and still, they are considered to be amid the most promising raw materials for other purposes, owing to their excellent environmental credentials, which include their ready availability, low cost, inherent biodegradability, low toxicity and their many versatile applications (Roseany *et al*, 2013).

The consumer and industrial interests in the development of eco-friendly materials have catapulted the environmentally benign agricultural resources as feedstock of the polymer industry. Today, due to interdisciplinary approaches through research and technological innovations in oleo-chemistry, biosciences, biotechnology and engineering, it is possible to design eco-friendly specialty chemicals from nature's abundant renewable resources.



Structure of a triglyceride, where R_1, R_2, and R_3 represent fatty acid chains

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Extraction of vegetable oils

Generally, extraction is the first step in the refining process of vegetable oils. Oils and fats are extracted from their original source (the seeds, fruits or other oil-bearing raw materials) using a variety of different methods. In some cases, the oil is extracted directly from the fruit by means of a simple mechanical press and used without the need for any further processing. This process is known as "cold pressing". For most oils however, the process is more complex. Modern oil mills extract oil using a combination of pressing, cooking and solvent extraction.

The seed/bean is cleaned and dried and foreign material is removed. Crushing used to be done between mill stones that later became steel rolls. Seeds with high oil content like rapeseed and sunflower seed are usually mechanically pressed in expellers after a preheating step in indirectly heated conditioners. The oil-bearing material is fed into one end of a cylinder where a power-driven worm conveyor forces the material to the other end of the cylinder and out against resistance. The pressure exerted in the process squeezes out the oil. Solvent extraction is used to separate oil from seeds/beans. The pre-processed seeds/beans are treated in a multistage counter current process with solvent until the remaining oil content is reduced to the lowest possible level. The mixture of oil and solvent is separated by distillation.

Oilseeds usually have the oil encased in minute "cells" that are deeply embedded in fibrous structures. Most oilseeds, therefore, require grinding or flaking to rupture the cells and heat treatment, sometimes cooking and drying, to help release the oil. Vegetable oilseed could either be processed by traditional crude method of crushing seed without delinting in undecorticated form or by scientific process which involves removal of linter, decortications, and separation of hull, expelling, solvent extraction and removal of oil. The chief disadvantages of solvent extraction are the high initial cost of the equipment and the fact that some oil seeds disintegrate under the influence of the solvent and consequently are difficult to handle. However, extraction with solvents constitutes the most efficient method for the recovery of oil from any oil-bearing material. The minimum oil content by which oil cake can be reduced by mechanical expression is approximately the same for all oil seeds, i.e, about 2-3%. Consequently, the oil unrecoverable by mechanical expression, in terms of percentage of the total oil, increases progressively as the oil content of the seed decreases. Substitution of solvent extraction for pressing methods increases the yield of oil (Tayde *et al*., 2011).

Composition and properties

Many naturally occurring fats are made up of fatty acids with chain lengths greater than 12 carbon atoms, the vast majority of vegetables and animal fats are made up of fatty acid molecules of more than 16 carbon atoms. The unsaturated fatty acids present in vegetable oils are oleic acid, linoleic acid and linolenic acid containing one, two and three double bonds between two carbon atoms respectively. The functionality present in vegetable oil is in terms of double bonds thus the C=C acts as a reaction site for chemical modification in vegetable oil (Bashar and Jumat, 2010).

Uses of vegetable oils

Oils extracted from plants have been used since ancient times and in many cultures. Archeological evidence shows that olives were turned into olive oil by 600BC and 4500 BC in present day Egypt (Linda, 2011). Many vegetable oils are consumed directly or indirectly as ingredients in food. A role that they share with some animal fats, including butter, ghee, lard, and schmaltz. The oils serve a number of purposes in this role:

- Shortening- to give pastry a crumbly texture.
- Texture- oils can serve to make other ingredients stick together less.
- Flavor- while less flavorful oils command premium prices, some oils such as olives, sesame, or almond oil, may be chosen specifically for the flavor they impart.

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- Flavor base-oils can also carry flavors of other ingredients, since many flavors are due to chemicals that are soluble in oil.

Secondly, oils can be heated and used to cook other foods. Oils suitable for this objective have high flash points. Such oils include the major cooking oils-soybean, rapeseed, canola, sunflower, safflower, peanut, cottonseed etc. Tropical oils, such as coconut, palm, and rice bran oils, are particularly valued in Asian cultures for high temperature cooking, because of their unusually high flash points. Industrially, vegetable oils are used as an ingredient or component in many manufactured products. They are used to make soaps, skin products, candles, perfumes and other personal care and cosmetic products. Some oils are particularly suitable as drying oils and are used in making paints and other wood treatment products. Dammar oil (a mixture of linseed oil and dammar resin), for example, is used almost exclusively in treating the hulls of wooden boats.

Vegetable oils are increasingly being used in the electrical industry as insulators, as vegetable oils are not toxic to the environment, biodegradable if spilled and have high flash points.

However, vegetable oils are less stable chemically, so they are generally used in systems where they are not exposed to oxygen, and they are more expensive than crude oil distillate. Synthetic tetra esters, which are similar to vegetable oils but with four fatty acid chains compared to the normal three found in natural esters, have high stability to oxidation and have found use as engine lubricants. It is used to produce biodegradable hydraulic fluid (Linda, 2011).

In the chemical industry, plant oils are used as an ingredient or component in many manufactured products, such as surfactants (soaps), lubricants, plasticizers, cosmetic products, monomers (e.g. dimer acids and polyols) and agrochemicals. In addition, they have been used for decades in paint formulation, as flooring materials and for coatings and resin applications (Roseany *et al*, 2013).

Non-edible oils use depends on the properties of the various fatty acids and fats. Some triglycerides are directly used as specialized lubricants. The many derivatives of fatty acids manufactures are used in surface coatings, lubricants etc, where the long hydrocarbon chain confers needed plasticity, surface activity, or lubricity. In some instances, e.g., drying oils, the reactive unsaturation of certain fatty acids is exploited and by means of catalyzed, oxidative polymerization, tough, flexible surface coatings are developed (Bashar and Jumat, 2010).

Some of the uses of vegetable oils are outlined below:

- Constituent of paint: Vegetable oils were used as the primary constituent in paints and coatings even during the days of cave paintings. Today, due to several environmental and health hazards cropping up from fossil fuel derived products, and fear of depletion of petroleum resources by the end of 21st century, the polymer chemists and technologists have reverted to the extensive utilization of vegetable oil derived materials in paints and coatings. Several vegetable oil-based materials have been developed and are tailored for various end use applications. Literature survey reveals that vegetable oils have enormous potential, significance and applications in the world of coatings.

- VO in coatings: VO and their derivatives find applications in coatings owing to their unique structural attributes.

- VO as corrosion inhibitors: Besides their use in coatings and paints as binders, VO and plant extracts have also been used as natural corrosion inhibitors. A recent review has appeared on the use of VO and other extracts for corrosion inhibition. Extracts from leaves and VO from seeds of several edible and medicinal plants have also been employed for anticorrosion behavior against alkaline and acid media as well as chloride ions. The anticorrosion is attributed to the presence of heterocyclic constituents such as alkaloids, flavonoids, tannins, cellulose, and others.

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- VO as polymeric coatings: Natural VOs are triacylglycerols of fatty acids. They contain suitable functionalities in their backbone such as double bonds, epoxies, hydroxyls, esters and other functional groups that can undergo several chemical reactions (Manawwer, *et al*., 2014).

A cracked coconut and coconut oil.

Extraction of coconut oil

Coconut oil can be extracted mainly through "dry" or "wet" process.

– Dry process: Dry processing requires that the meat be extracted from the shell and dried using fire, sunlight or kilns to create copra. The copra is pressed or dissolved with solvents producing the coconut oil and a high-protein, high-fiber mash. The mash is of poor quality for human consumption and is instead fed to ruminants.

– Wet process: the wet process uses raw coconut rather than dried copra, and the protein in the coconut creates an emulsion of oil and water. The more problematic step is breaking up the emulsion to recover the oil. This used to be done by prolonged boiling, but this produces a discoloured oil and is not economical. Modern techniques use centrifuges and pre-treatments including cold, heat, acids, salts, enzymes electrolysis, shock waves, steam distillation or some combination. Conventional coconut oil processors use hexane as a solvent to extract up to 10% more oil than produced with just rotary mills and expellers. They then refine the oil to remove certain free fatty acids to reduce susceptibility to rancidification (Hamid, *et al*., 2011).

Other methods of extraction are discussed below:

Cold extraction: Cold extraction is the term used for the extraction of coconut oil from coconut milk by breaking the emulsion without heating (Agarwal 2017). The high stability of the coconut milk emulsion needs the destabilization of coconut milk which can be done in three stages. In the first stage, cream is separated by the action of gravitational force resulting in two phases, the top phase with the creamy layer and the down phase with aqueous layer. The second stage is flocculation and clustering in which the oil phase moves as a group, and which does not involve the rupture of the interfacial film that normally surrounds. The third phase is the most critical phase. In the destabilization of coconut milk (Raghavendra, and Raghavarao, 2010), coalescence in this stage of interfacial areas is ruptured and reduced. This helps to join oil globules together (Sant'Anna, *et al*, 2003). This method appears more desirable due to elimination of solvents, refining, bleaching and deodorizing process, which reportedly may lower the investment cost and energy requirements, thus more environmentally friendly than the solvent extraction. Therefore, it can be carried out at home by anyone who is interested in producing their own natural oil. Even though the concept appears potentially attractive, however, the method yields comparatively low content of oil, which has discouraged its commercial use (Madhavan, *et al*, 2005). Cold extraction processes have been reviewed and can be presented under the following headings:

i. Chilling, Freezing and Thawing Method (Madhavan, *et al*, 2005 and Seneviratne, *et al*, 2009).

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ii. Centrifugation Method (Seneviratne, *et al*, 2009).

iii. Fermentation Method (Siddalingaswamy, *et al*, 2011 and Srivastava, *et al*, 2016).

iv. Aqueous Enzymatic Extraction Method (Srivastava, *et al*, 2016 and Siddalingaswamy, *et al*, 2011)

Hot Extraction Process: In Hot extraction processes, coconut oil is extracted from coconut milk by heating. Due to heating, the proteins of coconut milk are denatured, and the milk emulsion is destabilized. Extraction of the VCO is done by heating coconut milk at 100-120^{O}C for 60mins, until the water is completely evaporated. To extract the VCO from coconut milk, the protein is coagulated by slow heating in VCO cooker and releases the oil that is separated from pertinacious residue by filtering through muslin cloth and the remaining residue is further heated to remove more oil (Agarwal, 2017).

Types of coconut oil

- Virgin coconut oil (VCO): this can be produced from fresh coconut milk, meat or residue. Producing from the fresh meat involves either wet-milling or drying the residue and using a screw press to extract the oil. VCO can also be extracted from fresh meat by grating and drying it to a moisture content of 10-12%, then using a manual press to extract the oil. Producing it from coconut milk involves grating the coconut and mixing it with water, then squeezing out oil. The milk can also be fermented for 36-48 hours, the oil removed, and the cream heated to remove any remaining oil. A third option involves using a centrifuge to separate the oil from the other liquids. Coconut oil can also be extracted from the dry residue left over from the production of coconut milk.

- **Refined coconut oil**: this is also known as refined, bleached, and deodorized coconut oil. (RBD) It is usually made from copra, dried coconut kernel, which is pressed in a heated hydraulic press to extract the oil. This yields practically all the oil present amounting to more than 60% of the dry weight of the coconut. This "crude" coconut oil is not suitable for consumption because it contains contaminants and must be refined with further heating and filtering. Another method for extraction of coconut oil involves the enzymatic action of alpha-amylase, polygalacturonases, and proteases on diluted coconut paste. RBD is used for commercial food processing, cosmetic, industrial and pharmaceutical purposes.

- **Hydrogenated coconut oil:** RBD can further be processed into fully hydrogenated oil to increase its melting point. Since virgin coconut oils melt at 24^{O}C (76^{O}F), foods containing coconut oil tend to melt in warm climates. A higher melting point is desirable in these warm climates, so the oil is hydrogenated. The melting point of hydrogenated coconut oil is 36-40^{O}C (97-104^{O}F). In the process of hydrogenation, unsaturated fats (monounsaturated and polyunsaturated fatty acids) are combined with hydrogen in a catalytic process to make them more saturated. Coconut oil contains only 6% monosaturated and 2% polyunsaturated fatty acids. In the partial hydrogenation process, some of these are transformed into trans fatty acids.

– **Fractionated coconut oil:** Fractionated coconut oil provides fractions of the whole oil so that its different fatty acids can be separated for specific uses. Lauric acid, a 12-carbon chain fatty acid, is often removed because of its high value for industrial and medical purposes. The fractionation of coconut oil can also be used to isolate caprylic acid and capric acid, which are medium-chain triglycerides, as these are used for medical applications, special diets and cosmetics, sometimes also being used as carrier oil for fragrances (Wikipedia, 2012).

The following table shows the composition of fatty acid content of coconut oil:

The approximate concentration of fatty acids in coconut oil (midpoint of range in source):

Fatty acid content of coconut oil	
Type of fatty acid	Pct
Caprylic saturated C8	7%
Decanoic saturated C10	8%

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Lauric saturated C12		48%
Myristic saturated C14		16%
Palmitic saturated C16		9.5%
Oleic monounsaturated C18:1		6.5%
Other		5%
black: Saturated; *grey*: Monounsaturated; *blue*: Polyunsaturated		

Black Seed Oil

Black seed (*nigella sativa)*, (BSBI LIST, 2007) is known by many names; these include black caraway, Roman coriander, and black cumin, to name but a few. It belongs to the family of Ranunculaceae. It has a specific gravity of 0.9000-0.9350@25^{O}C, refractive index of 1.5010-1.5060@20.00^{O}C, and blends well with all essential oils. It is grown in many countries like Egypt, Turkey, India, Pakistan and Middle East. It is a native of South and Southwest Asia. The plant grows to 20-30cm (7.9-11.8in) tall, with finely divided, linear (but not thread-like) leaves. The flowers are delicate, and usually coloured pale blue and white, with five to ten petals. The black caraway fruit is a large and inflated capsule composed of three to seven united follicles, each containing numerous seeds which are used as spice, sometimes as a replacement for black cumin (Buniumbulbocastanum) (Heiss, 2005).

A picture of black seed flower adopted from the internet.

A picture of black seeds still in the pods adopted from the internet

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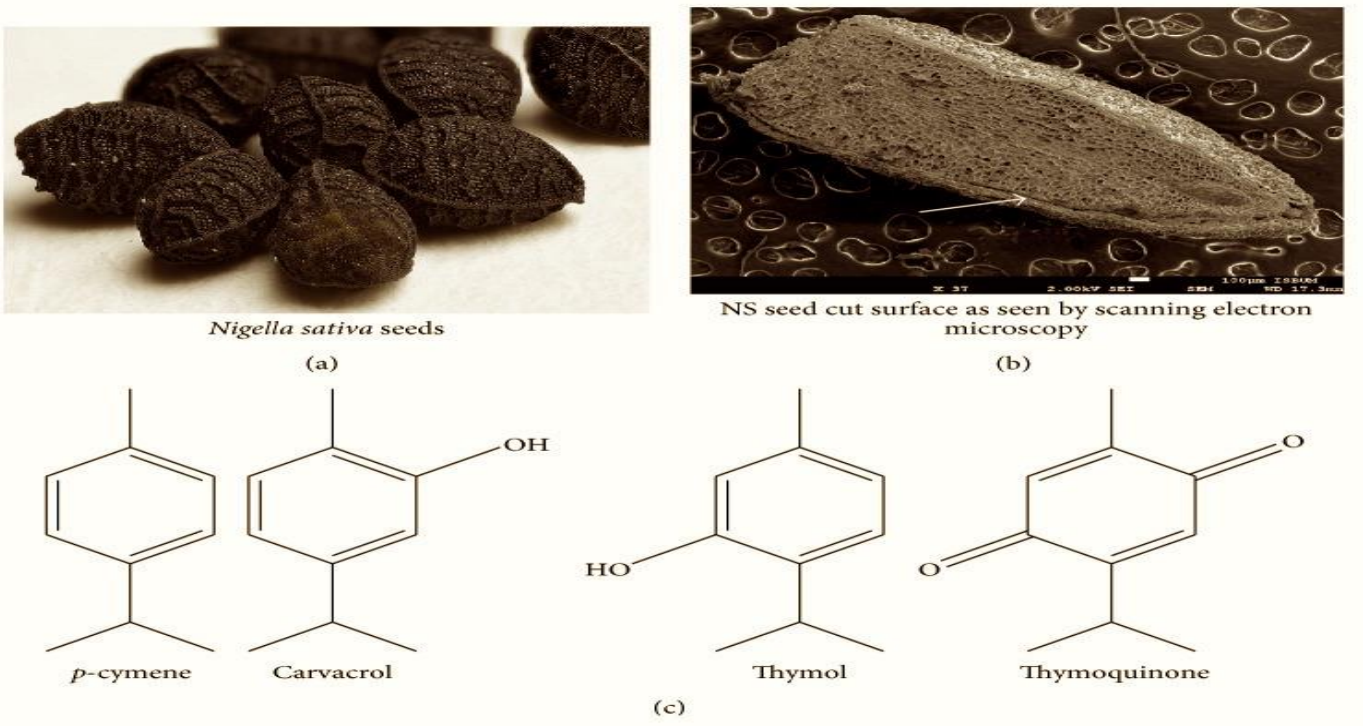
Picture of blackseeds

Macroscopically, seeds are small dicotyledonous, trigonus, angular, regulose-tubercular, 2-3.5mm×1-2 mm, black externally and white inside, odor slightly aromatic and taste bitter. Microscopically, transverse section of seed shows single layered epidermis consisting of elliptical, thick-walled cells, covered externally by a papillose cuticle and filled with dark brown contents. Epidermis is followed by 2-4 layers of thick walled tangentially elongated parenchymatous cells, followed by a reddish-brown pigmented layer composed of thick walled, rectangular elongated cells. Inner to the pigment layer, is present a layer composed of thick walled rectangular elongated or nearly columnar, elongated cells. Endosperm consists of thin walled, rectangular or polygonal cells mostly filled with oil globules. The powder microscopy of seed powder shows brownish black, parencymatous cells and oil globules. (Khare, (2004) and Warrier *et al.*, (2004).



A bottle of black seed oil

The seeds of Nigella Sativa are used as a spice in Indian and Middle Eastern cuisines. The black seeds taste like a combination of onions, black pepper and oregano. They have a pungent bitter taste and smell (Bharat, 2015).

The dry roasted nigella sativa seeds flavor curries, vegetables and pulses. It can be used as a “pepper” in recipes with pod fruit, vegetables, salads and poultry. In some cultures, black seeds are used to flavor bread products. It is also used as part of the spice mixture panchphoron (meaning a mixture of five spices) and most recognizably in naan bread (Bramen, 2011).

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Composition and chemical makeup

Nutritionally, the Nigella Sativa seed is comprised of approximately 21% carbohydrates and 35-38% fats (varies from country to country). It also contains numerous vitamins, mineral elements, and trace elements. The protein content of the black seed (Nigella Sativa) is comprised of fifteen different amino acids, including eight of the nine essential amino acids. Essential amino acids cannot be synthesized in our body in sufficient quantities and are thus derived entirely from our diet. Black seed oil is particularly rich in unsaturated and essential fatty acids (Linoleic and Linolenic acid) alpha-Linolenic acid (omega-3) and Linoleic acid (omega-6), are substances that cannot be produced in the body, and thus must be taken. The black seed oil is also a source of calcium, iron, sodium, and potassium. Required in small amounts by the body, these trace elements are essential for various enzyme functions (NABI BLACKSEED, 2014).

Detailed breakdown of what is in black seed oil:

Fatty acid	Percentage
Lauric acid	0.6
Myristic acid	0.5
Palmitic acid	12.5
Stearic acid	3.4
Oleic acid	23.4
Linoleic acid	55.6
Linolenic acid	0.4
Eicosadienoic acid	3.1
Non-terpenoid hydrocarcons	**Percentage**
n-Nonane	1.7
3-Methyl nonane	0.3
1,3,5-Trimethyl benzene	0.5
n-Decane	0.4
1-Methyl-3-propyl benzene	0.5
1-Ethyl-2,3-dimethyl benzene	0.2
n-Tetradecane	0.2
n-Hexadecane	0.2
Mono-terpenoid hydrocarbons	Percentage=26.9
α-Thujene	2.4
α-Pinene	1.2
Sabinene	1.4

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-Pinene	1.3
Myrcene	0.4
α-Phellandrene	0.6
p-Cymene	14.8
Limonene	4.3
y-Terpinene	0.5
Mono-terpenoid Ketones	Percentage=6
Fenchone	1.1
Dihydrocarvone	0.3
Carvone	4.0
Thymoquinone	0.6
Mono-terpenoid-ols	Percentage=2.7
Terpinen-4-ol	0.7
p-Cymene-8-ol	0.4
Carvacrol	1.6
Sesquiterpenoid hydrocarbons	Percentage=1.0
α-Longipinene	0.3
Longifolene 1408	0.7
Phenyl propanoid compounds	Percentage=46.1
Estragole	1.9
Anisaldehyde	1.7
trans-Anethole	38.3
Myristicin	1.4
Dill apiole	1.8

The Nigella Sativa oil contains conjugated linoleic (18:2) acid, thymoquinone, nigellone (dithymoquinone), melanthin, nigilline, and trans-anethole (Bharat. 2015).

Preliminary studies have investigated claims from traditional medicine that Nigella Sativa has efficacy as a therapy, mainly using the seed oil extract, volatile oil, and isolated constituent thymoquinone. One meta-analysis of clinical trials found weak evidence that Nigella Sativa has a short-term benefit on lowering systolic and diastolic blood pressure, and another found limited evidence that various extracts of black seed can reduce triglycerides, LDL and total cholesterol while raising HDL cholesterol (Sahebkar*et al.*, 2016 and Sahebkar*et al.*, 2016).

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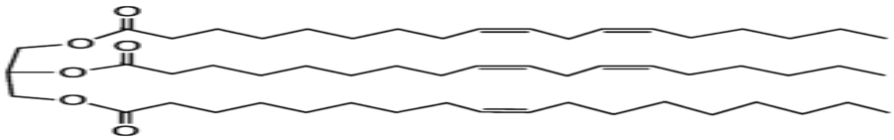
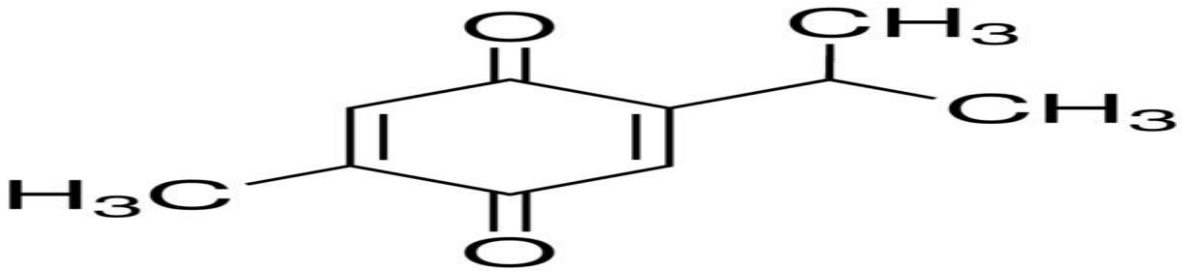


Structure of black seed oil

Nigella Sativa has been extensively studied for its biological activities and therapeutic potential and shown to possess wide spectrum of activities *viz.* as diuretic, antihypertensive, antidiabetic, anticancer and immunomodulatory, analgesic, antimicrobial, anthelmintics, analgesics and anti-inflammatory, spasmolytic, bronchodilator, gastroprotective, hepatoprotective, renal protective and antioxidant properties. The seeds of *N. sativa* are widely used in the treatment of various diseases like bronchitis, asthma, diarrhea, rheumatism and skin disorders. It is also used as liver tonic, digestive, anti-diarrheal, appetite stimulant, emmenagogue, to increase milk production in nursing mothers to fight parasitic infections, and to support immune system (Abdel-salam, (2012), Khaled, (2009), Assayed. (2010), Abdel-Zahar, *et al*., (2011), Boskabady.*et al*., (2010), and Goreja, (2003).

Most of the therapeutic properties of this plant are due to the presence of thymoquinone (TQ) which is a major active chemical component of the essential oil. Black seeds are also used in food like flavoring additive in breads and pickles because it has very low level of toxicity (Al-Ali, *et al*., (2008).



The chemical structure of thymoquinone (2-isopropyl-5-methyl-1,4-benzoquinone) (Majed, 2013).

Extraction of Black seed oil

Methods of extraction of seed oils are an effective factor in the properties of oils. Solvent extraction, for example, is deficient in selectivity and needs extreme heat, which could cause the degradation of the desired components (Pokorny and Korczak, 2001). The cold press extraction is the conventional method for oil extraction. It involves no and/or chemicals and is preferred by consumers concerned about natural and safe food (Kiralan, *et al*, 2014). However, this method affords low yields (Soto, *et al*, 2007), and the residual meal contains 10-12% oil content, which can eventually limit its uses in industries processing food (Zuniga, *et al*, 2003). Supercritical fluid extraction (SFE) is currently a technique among others used to extract plant oils and offers some favourable features over the traditional techniques that have been used in the oil industry (Akanda, *et al*, 2012). In general, SFE has been the method used to extract antioxidant compounds from NSO and exhibits a higher concentration of thymoquinone (Solati, *et al*, 2014).

The seeds of black cumin have length of about 0.3 cm each and have black colour. The seeds contain fixed and essential oils, alkaloids, proteins and saponin. Most of the biological activity has been attributed to the major components of the essential oil – thymoquinone– (24.5-57%), ρ-cymene (10.7-40.3%), α-thujen (1.9-8.2%), carvacrol (2,2-4.5%), 4-terpineol (1.9-4.5%) (Burits, 2000).

Sorrel oil

Common sorrel or garden sorrel (Rumex acetosa), often simply called sorrel, is a perennial herb in the family Polygonaceae. Other names for sorrel include spinach dock and narrow-leaved dock. It is a common plant in grassland habitats and is cultivated as a garden herb or salad vegetable (pot herb) (Wikipedia).

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Garden sorrel plant showing the leaves, fruit and flower

Sorrel is a slender herbaceous perennial plant about 60cm (24in) high, with roots that run deep into the ground, as well as juicy stems and edible, arrow-shaped (sagittate) leaves. The leaves, when consumed raw, have a sour taste. The lower leaves are 7 to 15cm (2.8 to 5.9in) in length with long petioles and a membranous ocrea formed of fused, sheathing stipules. The upper ones are sessile and frequently become crimson. It has whorled spikes of reddish-green flowers, which bloom in early summer, becoming purplish (Blamey, *et al.,* 2003; and Stace, 2010). The specie is dioecious, with stamens and pistils on different plants (Stace, 2010). Common sorrel has been cultivated for centuries. The leaves may be pureed in soups and sauces or added to salads; they have a flavor that is similar to kiwifruit or sour wild strawberries. The plant's sharp taste is due to oxalic acid, which is mildly toxic. In northern Nigeria, sorrel is known as yakuwa or sure (pronounced suuray) in Hausa or karassu in kanuri. It is also used in stews usually in addition to spinach. In some Hausa communities, it is steamed and made into salad using kuli-kuli (traditional roasted peanut cakes with oil extracted), salt, pepper, onion and tomatoes. The recipe varies according to different levels of household income.

Sorrel seeds

Hibiscus sabdariffa plant

Specie of sorrel is the *Hibiscus species* which belongs to Malvaceae family and are native to Southern Asia and West Africa respectively. These two plants can grow well under such adverse climate because of their low

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moisture demands, fertility requirements and tolerance to high temperatures and are a drought-resistant warm season annual or biennial, herbaceous plants growing to about 16-20ft tall with a woody base in a wide range of soils (Kaushik, *et al*, 2007). The stems are 1-2cm in diameter and often but not always branched. The leaves are 10-15cm long, variable in shape with leaves near to the base of the stems. The flowers are 15-18cm in diameters, white yellow or purple, and when white or yellow the centre is still dark purple. The fruits are capsules of 2cm in diameter containing around 20-25 seeds (Ming and Brad, *et al*, 2012).

Sorrels are quick growing crops, and their seeds become capable of having their oil extracted after 4-5 months of plantation depending on the soil quality and rainfall. The annual yield of these two plants is in the range from 0.5 to 12 tons and can also grow at low and high-altitude areas that have an average annual temperature above 20^{O}C and can tolerate slight frost. The cultivation of these two plants is successful in the tropics with the annual rainfall of 2500,000 mm (Rashmi and Naik, 2015). The marginal soil quantities with a low nutrient content are sufficient for growing of Hibiscus cannabinus and Hibiscus sabdariffa plants.

Hibiscus cannabinus and Hibiscus sabdariffa the plants with multifunctional properties with many attributes and considerable potential. The various parts of these two plants have many useful applications (Satheesh, *et al*, 2009). The stems are used in making rope, cordage, canvas, sacking, carpet backing, fishing nets, jute fibers and domestic purposes. Leaves are used as vegetables in preparing salad and for cooking purposes. Seeds contain oil which is used as good lubricant oil and making soap, flowers or calyces are used in preparing tea. And can be used as medicine in treating diuretic, mild laxative, relieve dysuria, bruises, fever, puerperium, anemia, fatigue, blood and throat disorders, treating acidity and treatment for cardiac and nerve diseases and cancer. The oil cake, a byproduct remaining after the extraction of oil can be used in the form of food for cattle and as an organic fertilizer (Rashmi and Naik, 2015).

A study was conducted on the seed oil of two Hibiscus species i.e. Hibiscus cannabinus and Hibiscus sabdariffa by Rashmi and Naik, 2015. The two species were analyzed to establish their physicochemical properties and fatty acid using standard methods. The oil was extracted using hexane ($40\text{-}45^{O}$) by Soxhlet apparatus and fatty acid profile was determined using Gas chromatography (GC) analyzer. The study showed that, *Kenaf* and *roselle* had golden yellowish oil color and oil content was found to be 24% (*Kenaf*) and 22% (*Roselle*). The acid value (3.02: 2.80 mg KOH/g), specific gravity (0.88:0.86), refractive index (1.46: 1.45), moisture content (1.88: 1.46%), viscosity (53.00:52.70mPas), FFA (0.77: 0.65%) and saponification values (172:170mgKOH/g) were higher in *Hibiscus cannabinus* when compared to that of *Hibiscus sabdariffa*, whereas iodine value (133:177gI/100g), peroxide value (13.3: 26.6meq/kg) and Unsaponifiable matter (0.052: 0.85%) were lower in *Hibiscus cannabinus* when compared to that of *Hibiscus sabdariffa*. Both plant seed oils were rich in oleic and linoleic acids. The seed oil with highest amount of polyunsaturated fatty acids can find an application in surface coating industries bio lubricant base oil applications; whereas the high amount of monosaturated fatty acid can find an application as a biodiesel feedstock. The evaluation of fatty acid composition using gas chromatography revealed that *Hibiscus cannabinus* contained higher amounts of oleic acid (28.91%) and linoleic acid (38.49%) and significantly lower amounts of stearic (3.96%) and palmitic acid (20.75%) than *Hibiscus sabdariffa* seeds (25.16%, 44.72%, 18.52% and 4.31% respectively). Hence, the two plants of *Hibiscus species* seed oil have a great potential for industrial applications such as in paint and surface coatings, bio-lubricant and production of biodiesel (Rashmi and Naik, 2015).

According to Vangalapati*et al.*, 2014, the sorrel seed oil is rich in unsaturated fatty acids and is also a rich source of lipid soluble antioxidants, particularly γ-tocopherol. The physico chemical parameters of Roselle seed oil is summarized as acidity, 2.24%; peroxide index, 8.63 meq/kg; extinction coefficients at 232(k232) and 270 nm (k270), 3.19 and 1.46, respectively; oxidative stability, 15.53 h; refractive index, 1.477; density, 0.92kg/L; and viscosity, 15.9cP. Roselle seed oil belongs to the linoleic/oleic category, its most abundant fatty acids being C18:2(40.1%), C18:1(28%), C16:0(20%), C18:0(5.3%), and C19:1(1.7%). Sterols include β-sitosterol (71.9%),campesterol (13.6%), Δ-5 avenasterol (5.9%), cholesterol (1.35%), and clerosterol (0.6%). Total tocopherols were detected at an average concentration of 2000 mg/kg, including α-tocopherol (25%), γ-tocopherol (74.5%), and δ-tocopherol (0.5%). By the physicochemical characteristics of the oil, it was shown that the sorrel seed oil can be used as edible oil and as an industrial feedstock (Vangalapati*et al*. 2014).

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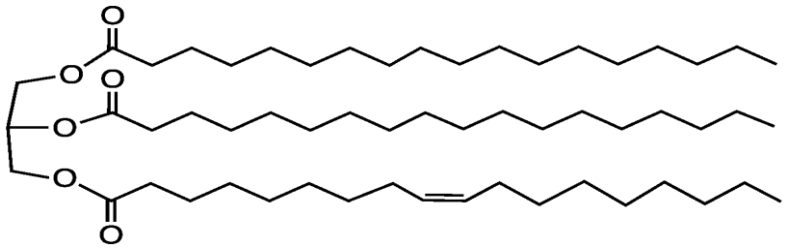

Structure of sorrel seed oil

Sorrel is cultivated in both tropical and subtropical regions for its popular edible calyces, stem fibre leaves and seed (Babajide *et al*, 2004; Mahadevan *et al* 2009). It is said to be of Asia or tropical African origin, its native being of India and Malaysia. Sorrel can be found in tropical areas such as the Caribbean, Florida, Central America, India, Africa, Brazil and the Philippines. It is known as a promising crop which has several uses and prospects for industrial potential. According to Akindahunsi and Olaleye (2003) sorrel is considered to be one of the most important and popular medicinal plants and it has several properties such as antiseptic, aphrodisiac, cholagogue, digestive and stomachic. It has not only won itself a household name but has also become known throughout the World for its usefulness. In Jamaica, the plant is popular at Christmas where emphasis is placed on its drink making properties. It is also a popular drink in Trinidad and Tobago and Africa.

Drink made from sorrel

There are many versatile ways in which sorrel can be used. These include its medicinal, cosmeceutical and traditional uses. Sorrel has a lot of uses. Traditionally, it has a lot of potential as an industrial crop which is now considered one of the competitive beverages in the World (Schipper, 2000). Seeds of this plant have been found to be a good source of protein and though somewhat bitter, it has been grounded for human consumption in Africa (Halimatul*et al*, 2007).

Medicinally, according to Joshi and Parle (2006), it possesses antihypertensive, antioxidant, anti-cancer, anticlastrogenic, hypolipidaemic, hepatoprotective, anti-stress, antispasmodic, diuretic and antidiarrheal activities. Morion (2008) observed that Hibiscus Sabdariffa tea, consumed daily, can reduce one's blood pressure to up to 13.2%. Garner-Wizard, Minigh and Webb (2007) conducted similar research which showed that Hibiscus Sabdariffa is just as effective as Lisinopril (a drug used to lower hypertension). A similar result was obtained when compared with another hypertension drug called captopril. Among the 70 persons studied, their blood pressure reduced to 10% in 79% of those who drank Hibiscus Sabdariffa tea versus 84% of those who used such drugs (Phytomedicine, 2004).

Since 2002, the scientific community in Jamaica and beyond was enthralled by local researchers at the Northern Caribbean University (NCU). As reported by Silvertorch.com (2011), Juliet Penrod demonstrated the curing effect of Hibiscus Sabdariffa and garlic on cancer cells. Their findings showed that when liver cancer cells were treated with Hibiscus Sabdariffa extract, their survival and effectiveness were severely curtailed even to the point of death. An array of researchers have concluded that Hibiscus Sabdariffa contains potent antioxidant principles (Ologundudu*et al.* 2009b). This makes it an effective essential tool to fight cancer (Northern Caribbean University, 2002). Anokwuru*et al* (2011) alluded to the fact that the calyxes are rich in phenolic compounds. This according to them has anti-bacterial, antiviral and antifungal actions in the plant

itself and by virtue of being hydrogen donating antioxidants, they protect against oxidative stress which can adversely affect the health of plants as well as humans. Josiah *et al*, (2010); Mahadevan *et al*, (2009) in support of this point stated that the calyxes are rich in phenolic compounds with marked protocatechuic acid which may have diuretic and choleretic effects decreasing the viscosity of the blood, reducing blood pressure and stimulating peristalsis.

Hibiscus Sabdariffa also has cosmeceutical uses. According to the Jamaica Science Research Council (2002), sorrel can be used as a cosmeceutical. Asher, (2005) stated that the Science research council has developed a sorrel care line of cosmetics made from the oil extract of the sorrel seed. The cosmetics included body wash, shampoos, hand and body lotions and eye cream. "The cosmeceutical nature of sorrel, where the herbs are used into a cosmetic base, has a lot of potential. Asher in her article further outlined that according to Diane Robinson, nutritionist, sorrel is good for its anti-ageing effects. It is high in vitamin C, thiamine, amino acids, and has antioxidant properties.

Hibiscus Sabdariffa has antiphlogistic and antioedema properties. These activities are probably due to the high level of mucilage, which allows to advise herbal teas for the treatment of eczemas and some allergies. Moreover, herbal tea lotion improves oily or devitalized skin, promotes hair growth and is used to treat abscesses (Burgundy Botanical Extracts, 2007).

Mahogany oil

Mahogany is known in the English language as African mahogany, and dry zone mahogany, it is known as madachi in the Hausa language, ono in the Igbo language and ogonwo in the Yoruba language (Orwa, *et al*, 2009).

Mahogany is a kind of wood that is straight-grained, reddish-brown timber of three tropical hardwood species of the genus swietenia, indigenous to the Americas (Bridgewater, 2012) part of the pantropical chinaberry family, Meliceae. The three species are:

1. Honduran or big-leaf mahogany (Swietenia macrophylla), with a range from Mexico to southern Amazonia in Brazil, the most widespread species of mahogany and the only true mahogany species commercially grown today (Bridgewater, 2012).

2. West Indian (Simon, *et al*, 2011) or Cuban mahogany (Swietenia mahogany), native to southern Florida and the Caribbean, formerly dominant in the mahogany trade, but not in widespread commercial use since World War II (Bridgewater, 2012).

3. Swietenia humilis, a small and often twisted mahogany tree limited to seasonally dry forests in Pacific Central America that is of limited commercial utility (Bridgewater, 2012). Some botanists believe that S. humilis is a mere variant of S. macrophylla (Bridgewater, 2012).

While the three Swietenia species are classified officially as "genuine mahogany", other Meliaceae species with timber uses are classified as "true mahogany". (Only the Swietenia species can be called "genuine mahogany"). Some may or may not have the word mahogany in their trade or common name. Some of these true mahoganies include the African genera Khaya and Entandrophragma; (Bridgewater, 2012) Chinese mahogany, Toona sinesis; (Christophe, 2006) Indonesian mahogany, Toona sureni; (Michel, 2005) etc.

Mahogany is a commercially important lumber prized for its beauty, durability, and colour, and used for paneling and to make furniture, boats, musical instruments and other items. The leading importer of mahogany is the United States, followed by Britain; (Bridgewater, 2012) while the largest exporter today is Peru, which surpassed Brazil after that country banned mahogany exports in 2001 (Donald, 2011).

Sky fruit is the fruit of mahogany tree (Swietenia macrophylla) that grows in the majority of Asian countries. This fruit is also named "buahtunjuklangit" in the Malay language. It gets its unique name due to the way it hangs on the tree. Unlike most fruits, which hang downward, sky fruit hangs upward and its stalk points to the

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sky. In almost all countries where mahogany grows, sky fruit is commonly used as herbal remedy that helps improve blood circulation, treat diabetes, and cure impotence (Khare, *et al*, 2012). Although scientific evidence affirming its efficacy as a herbal remedy is still weak, many people have already used it, and anecdotal reports have recorded the health benefits of the fruit. Besides, the revelation of various substances that the fruit contains may give reliable scientific clues of how efficacious the fruit is and how numerous sky fruit benefits are (skyfruitseed.com).

Picture of mahogany tree showing the fruit

Picture of mahogany fruit

The broken pods of mahogany fruit

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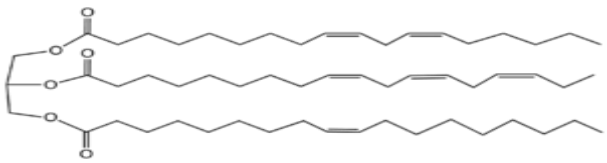
The mahogany fruit capsule is about 5–10 cm long, 3–6 cm in diameter, ovoid or oblong, 5-celled, splits from base to apex, valves thick, woody, surface coriaceous when mature. Each of the capsule contains about 35–45 seeds, brownish in color, 4–5 cm long, compressed, crested and extended into a wing at the point of attachment.

The unshelled mahogany seeds

Shelled mahogany seeds

The mahogany seed oil which is extracted from the seeds of mahogany fruit had its fatty acid composition analyzed by Gas Chromatography and a total of 48 compounds were identified. The major constituents of the methylated fatty esters were linoleic acid (26.00%), elaidic acid (24.39%), stearic acid (14.32%), palmitic acid (12.97%), 10-methyl-10-nonadecanol (5.24%), ecosanoic acid (2.48%), 3-heptyne-2,5-diol, 6-methyl-5-(1-methylethyl) (2.03%) octadecanoic acid, 9,10,12-trimethoxy (1.90%); 1,3-dioxalane, 4 ethyl-4-methyl-2-pentadecyl (1.89%) and 2-furapentanoic acid (1.03%). It is evident from this study that the oil can be considered as a good source of unsaturated fatty acids. The oil is bitter in taste and considered as a moderate drying oil, which can be useful in different chemical industries for soap and dying. The degree of unsaturation of MSO is very low. Oleic acid is the only unsaturated fatty acid in the composition of MSO, and its relative percentage is less than 1% (Rana, *et al*, 2015).

Okieimen and Eromosele (1999) stated that mahogany (Khaya senegalensis) seed oil contains 52.5 – 67% oil content. This is a reasonable yield and according to Jibrail and Kaet (2013) this yield is desirable for biodiesel production.



Structure of mahogany seed oil

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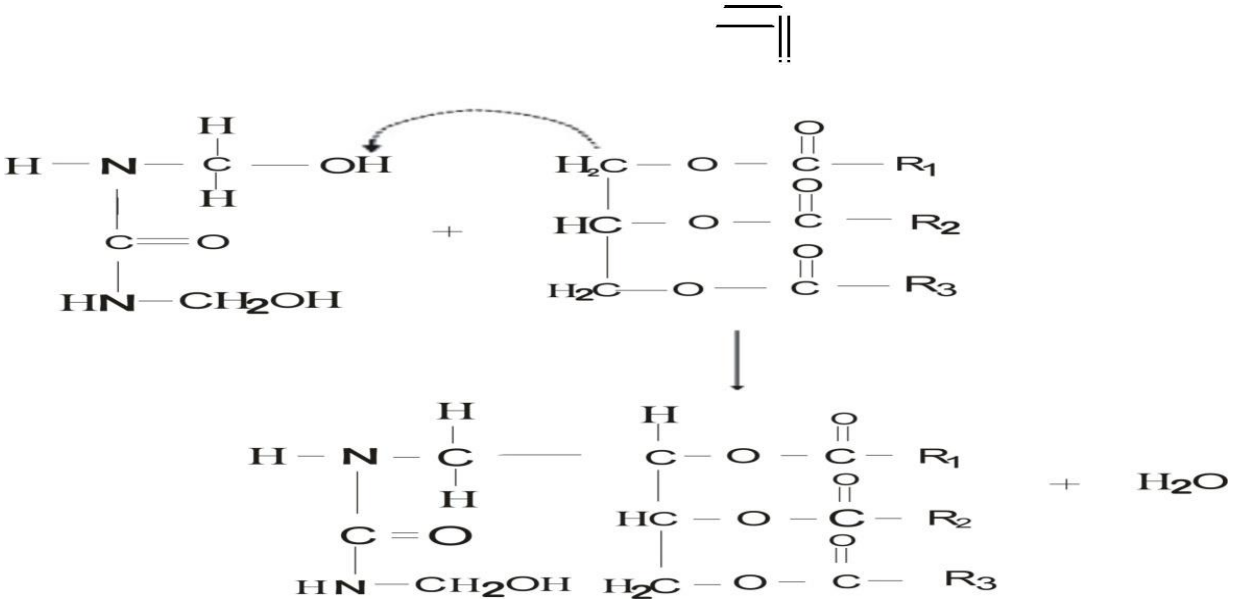
The following gives a breakdown on the physico-chemical properties of mahogany seed, according to Bamaiyi, *et al* (2007):

Parameters	Results
Colour	Brown
Moisture	24.60%
Specific gravity at $30^{O}C$	0.9334
Acid value	10.92
Free fatty acid (FFA)	5.49% (as oleic acid)
Saponification value	191.27
Iodine value	94.4
Unsaponifiable matter	1.49%
Oil (dry basis)	53.75%
Polenske value	0.35

Mahogany seed oil can be used in the energy sector for the production of biodiesel (Rana, *et al*, 2015 and Bamaiyi, *et al*, 2007). It can also be used in the production of methylester (Danbature, *et al*, 2015). It is used as fumigants or contact insecticides to protect grains, especially legumes against storage insects (Bamaiyi, *et al*, 2007). Used for cooking as well in cosmetics.

Chemical interaction between DMU and Vegetable oil.



METHOD

Introduction

This chapter deals with the method that was adopted in carrying out the study. Specifically, the procedure employed in Evaluation of Composites from Dimethylol Urea/Hydroxylated Vegetable Oils for Possible Application for Water Resistant Emulsion Paint. It contains the design, setting, sampling techniques, sample size determination, and method of data collection.

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Design

The study will employ an experimental research design to evaluate the properties of composites from dimethylol urea/hydroxylated vegetable oils for possible application in water-resistant emulsion paint.

Setting

The study will be conducted in a laboratory setting, where the synthesis and characterization of the composites will take place.

Sampling Techniques

The study will use the following samples:

i. Vegetable oils (coconut oil, black seed oil, mahogany oil, and sorrel oil) extracted from their respective seeds.

ii. Dimethylol urea (DMU) synthesized from urea and formaldehyde.

iii. Hydroxylated vegetable oils (HVOs) prepared through epoxidation and hydroxylation of the extracted vegetable oils.

iv. Composites of DMU/HVOs prepared through copolymerization.

Method of Data Collection

i. Extraction of vegetable oils from their respective seeds.

ii. Synthesis of DMU and HVOs.

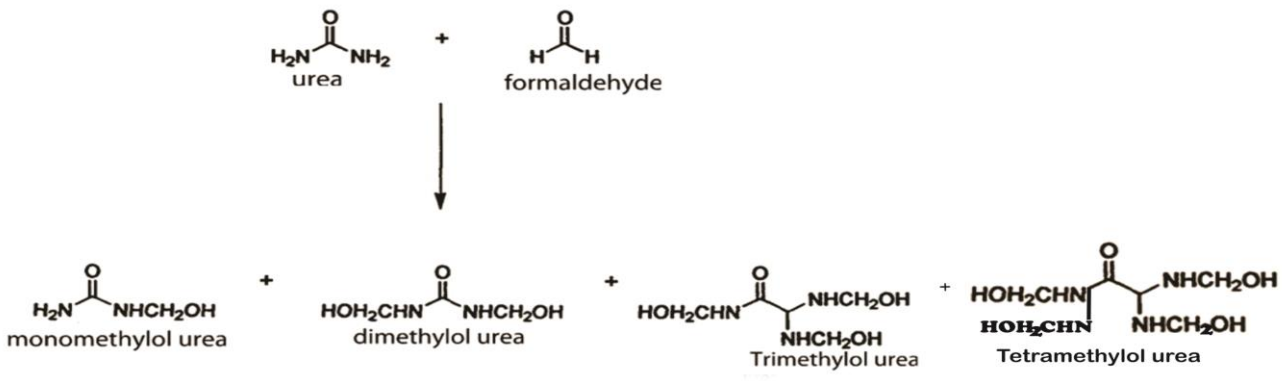
iii. Copolymerization of DMU and HVOs.

iv. Characterization of the resulting composites using various techniques (e.g., FTIR, NMR, DSC).

v. Formulation of emulsion paint using the composite resin as a binder.

vi. Evaluation of the physical properties of the formulated emulsion paint (e.g., adhesion, flexibility, water resistance).

RESULTS AND DISCUSSIONS



INTRODUCTION

This chapter presents results on the synthesis and characterization of Dimethylol Urea/Hydroxylated Vegetable Oil copolymers and their evaluation as binders for water-resistant emulsion paint. The oils used were Coconut

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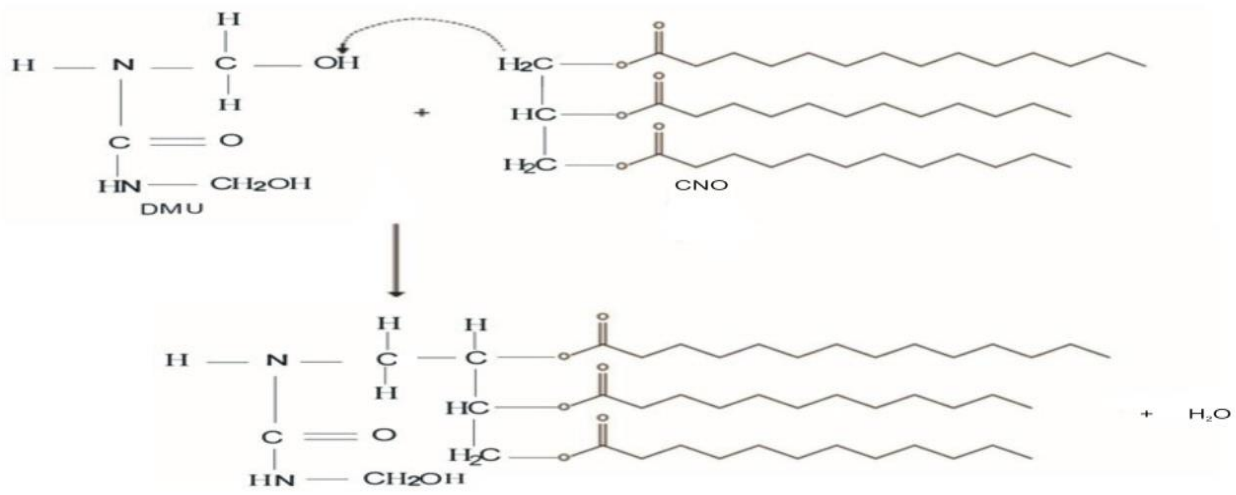
Oil (CNO), Sorrel Oil (SO), Mahogany Oil (MSO) and Black Seed Oil (BSO). The results are discussed in line with the objectives: extraction, synthesis, modification, copolymerization, characterization, paint formulation and evaluation.

The addition reaction of urea with formaldehyde produced mono-, di-, and trimethylol urea derivatives, which served as the base resin for copolymerization (Thomas et al., 2011).

Effect of CNO Concentration on DMU/CNO Copolymer Composite.

Reaction of DMU with CNO



FTIR spectral analysis

DMU exhibited broad O-H and N-H stretches at 3700-3300 cm^{-1} and C=O. CNO showed O-H at 3469 cm^{-1} due to water absorption, C=O of ester at 1746 cm^{-1}, C-H bending at 1453 cm^{-1}, and S=O at 1363 cm^{-1}. Absence of peaks at 3007 and 1655 cm^{-1} confirmed unsaturation in CNO (Cheman & Rohman, 2013).

In the DMU/CNO blend, peaks for O-H, N-H, C=O, C-H and C=C were retained. The presence of free O-H at 3717 cm^{-1} and C-O of aldehyde indicated that hydroxylation did not destroy CNO functional groups. This confirms successful copolymerization and formation of a new polymer network with 1,2,3-trisubstituted structures.

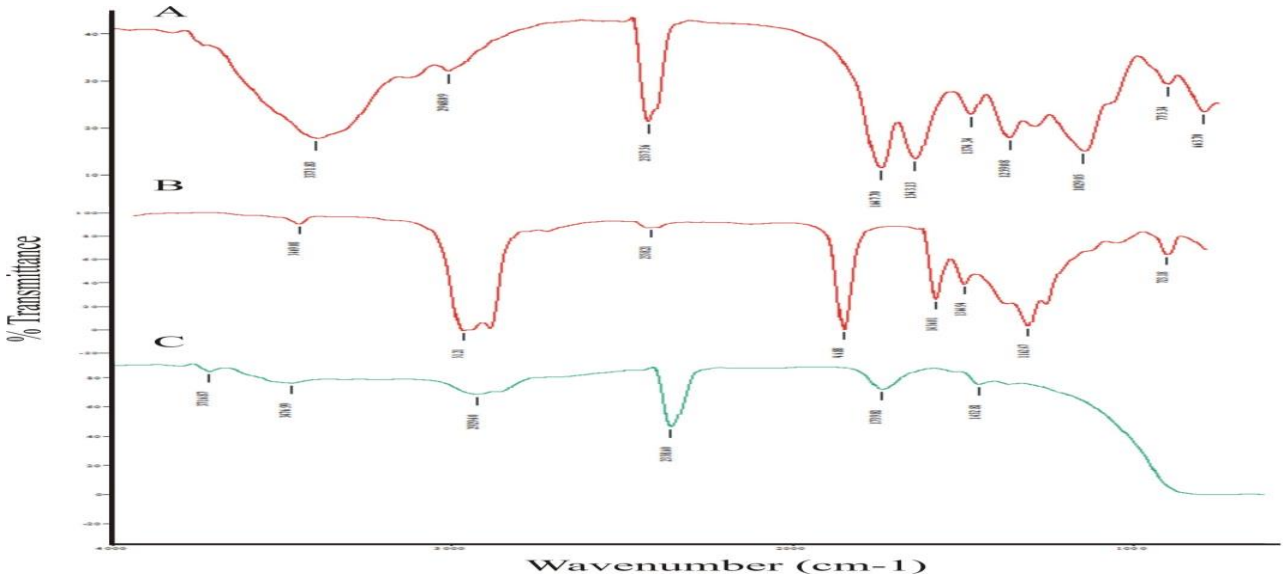


Figure 4.1.3

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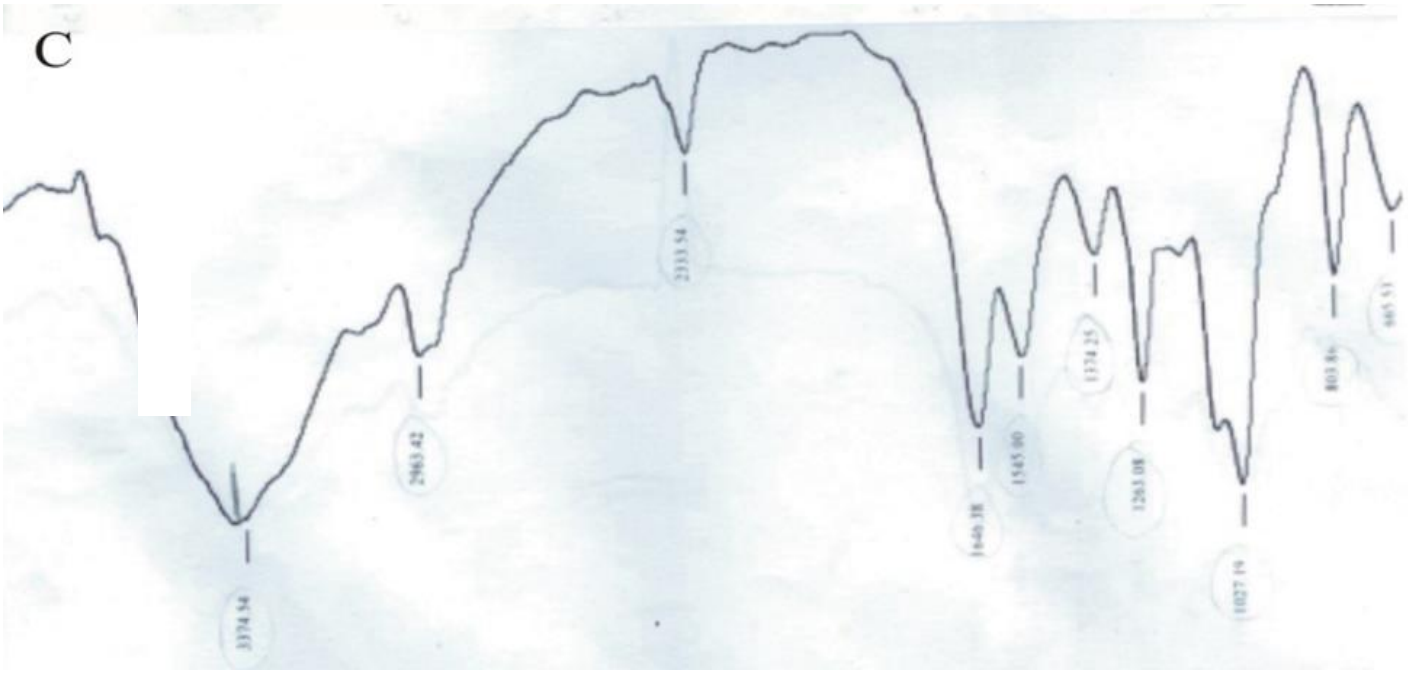
A – FTIR Spectra of DMU, B – FTIR Spectra of CNO, C – FTIR Spectra of DMU/CNO.



Effect of CNO concentration on the viscosity of DMU/CNO copolymer composite.

Figure 4.1.4 shows viscosity increased with CNO loading up to 50ml then decreased. The initial rise is due to increase in molecular weight and chain entanglement (Afzal et al., 2013). Beyond 50ml, overloading caused polymer dissociation into oligomers, reducing viscosity (Osemeahon& Dimas, 2014).

Viscosity is critical in coatings as it controls flow, leveling, sagging, film formation and adhesion (Osemeahon & Barminas, 2007). The optimum viscosity at 50ml CNO suggests good application properties.

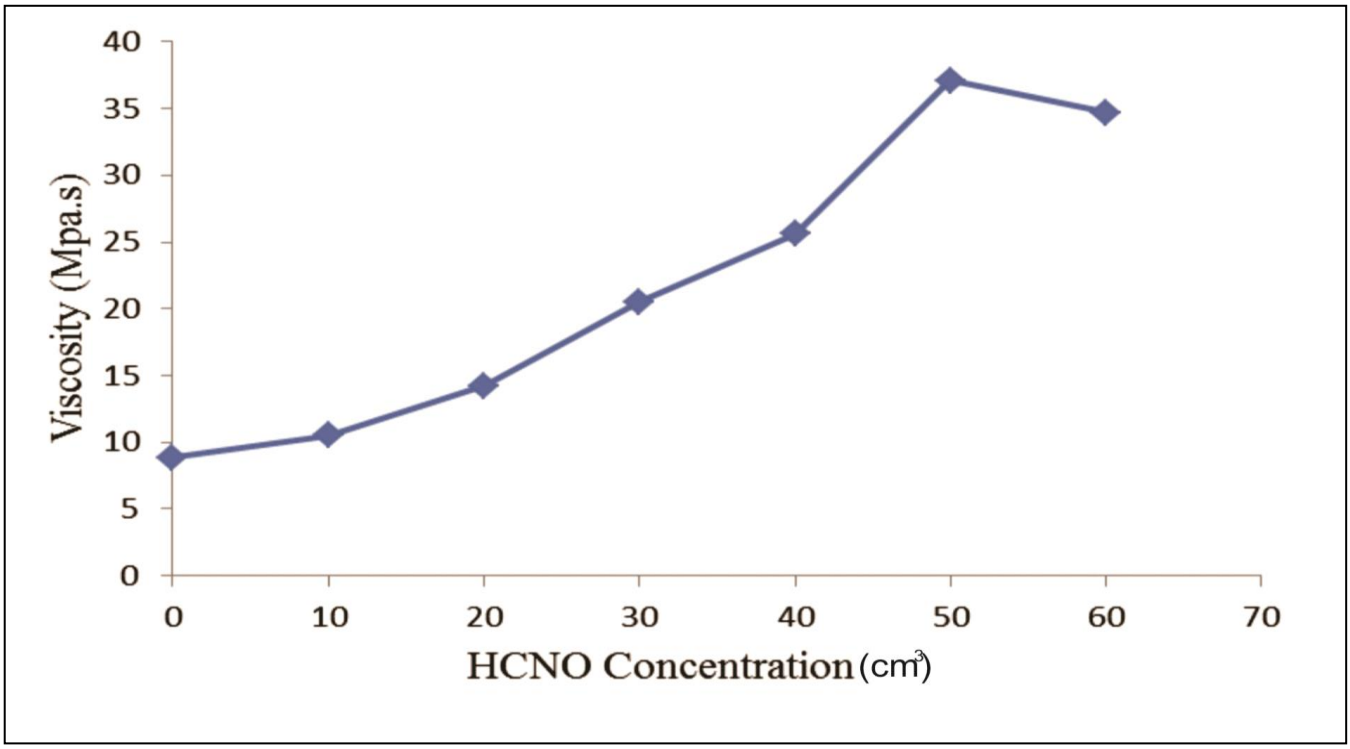


Fig.4.1.4. Effect of CNO concentration on the viscosity of DMU/CNO copolymer composite.

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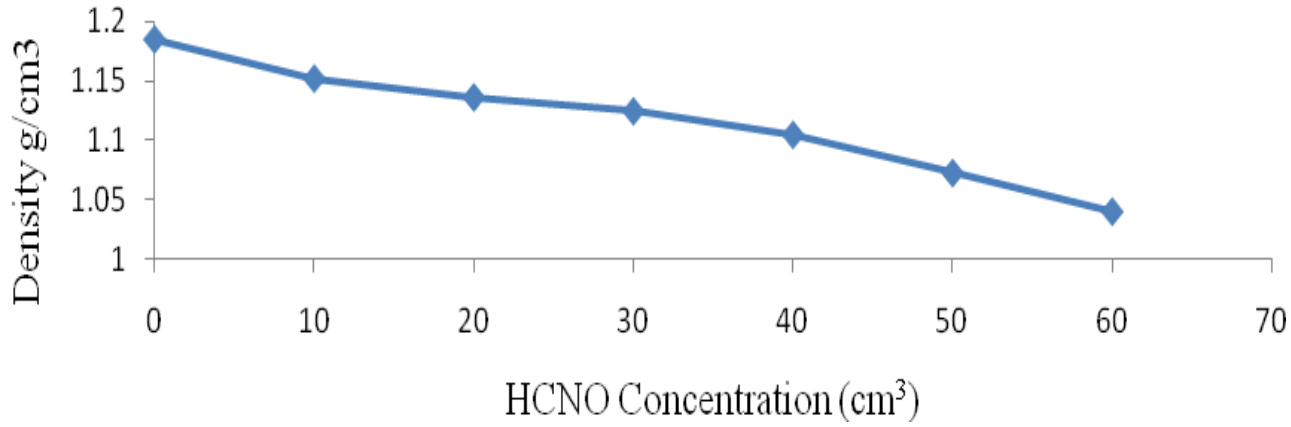
Effect of CNO concentration on the density of DMU/CNO copolymer composite.

Figure 4.1.5 shows a gradual decrease in density with CNO loading. This is due to lower density of CNO and less efficient molecular packing as soft segments increased (Osemeahon & Dimas, 2014). Density affects pigment dispersion, flow and brushability in the coating industry (Kazys & Rekuviene, 2011).



CNO Concentration (cm^3)

Fig. 4.1.5. Effect of CNO concentration on the density of DMU/CNO copolymer composite.

Effect of CNO concentration on the gel-time of DMU/CNO copolymer composite.

Figure 4.1.6 shows gel-time increased with CNO loading and became steady after $60cm^3$. This is attributed to increased molecular weight and viscosity (Osemeahon & Dimas, 2014). Gel-time, or pot life, determines working time and drying rate of paint (Menkiti & Onukwuli, 2011). Longer gel-time at higher CNO gives better storage stability.

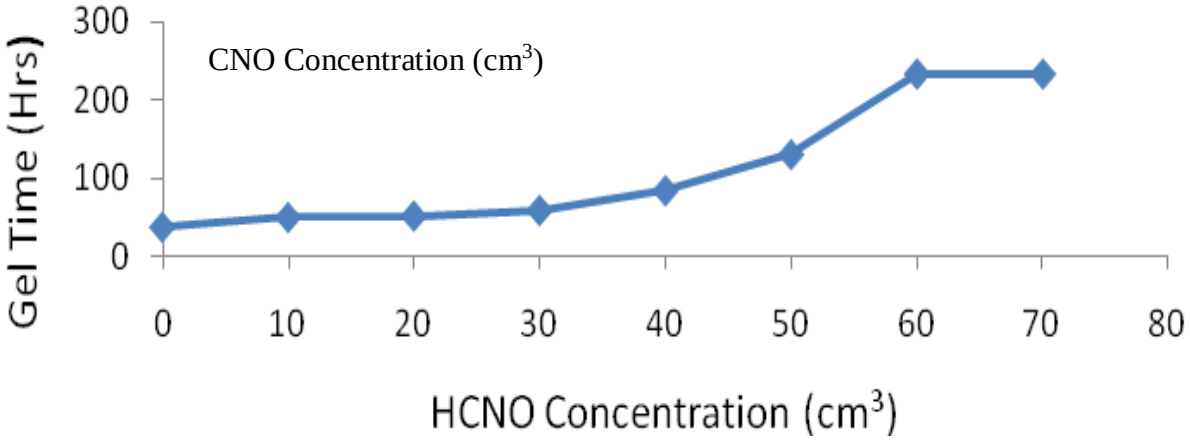


Fig. 4.1.6. Effect of CNO concentration on the gel-time of DMU/CNO copolymer composite.

Effect of CNO concentration on the turbidity of DMU/CNO copolymer composite.

Figure 4.1.7 shows turbidity increased with CNO concentration. Pure DMU is clear, but addition of CNO caused light scattering due to formation of large aggregates (Osemeahon & Dimas, 2014). Turbidity relates to gloss and optical properties of the binder.

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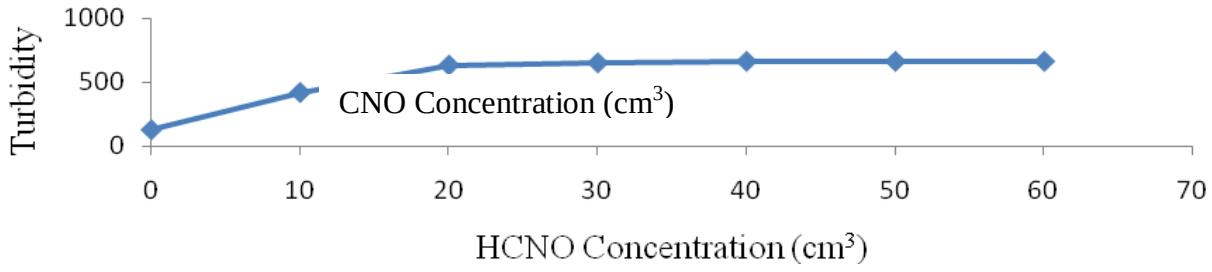

Fig. 4.1.7. Effect of CNO concentration on the turbidity of DMU/CNO copolymer composite.

Effect of CNO concentration on the refractive index of DMU/CNO copolymer composite.

Figure 4.1.8 shows refractive index increased with CNO loading. This is due to increased crystallinity and molecular features (Jain, 2008). Higher refractive index improves gloss, which is important for decorative emulsion paint to compete with oil paint (Kaygin & Akgun, 2009).

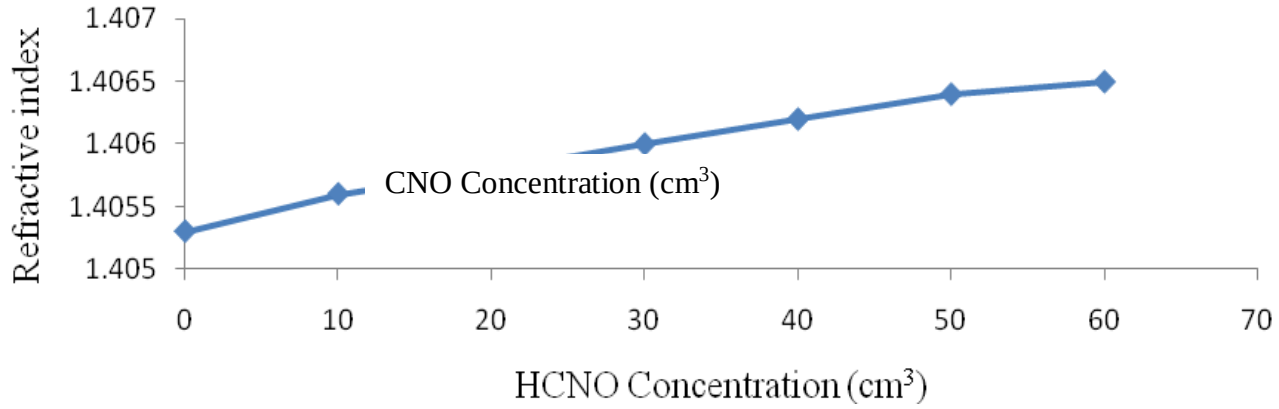

Fig.4.1.8. Effect of CNO concentration on the refractive index of DMU/CNO copolymer composite.

Effect of CNO concentration on the formaldehyde emission of DMU/CNO copolymer composite.

Figure 4.1.9 shows formaldehyde emission decreased as CNO increased. This is due to reduced DMU content and the hindrance effect of CNO, which disrupts DMU matrix continuity (Lee et al., 2011). Reducing formaldehyde emission addresses health concerns like sick building syndrome associated with urea-formaldehyde resins (Salthammer et al., 2010; Park et al., 2010).

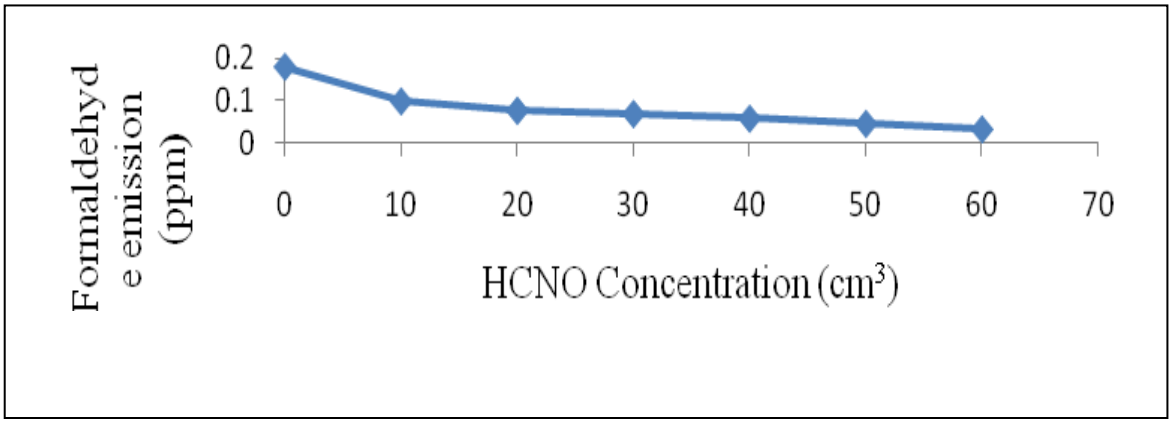

Fig.4.1.9. Effect of CNO concentration on the formaldehyde emission of DMU/CNO copolymer composite.

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Effect of CNO concentration on the melting point of DMU/CNO copolymer composite.

Figure 4.1.10 shows melting point decreased from 0-40ml CNO due to increased flexibility, then sharply increased at 50ml due to higher crosslink density, and decreased again at 60ml due to dissociation. Optimal hardness was at 50ml (Osemeahon& Dimas, 2014). Melting point relates to thermal resistance and brittleness of binders (Afsoon et al., 2011).

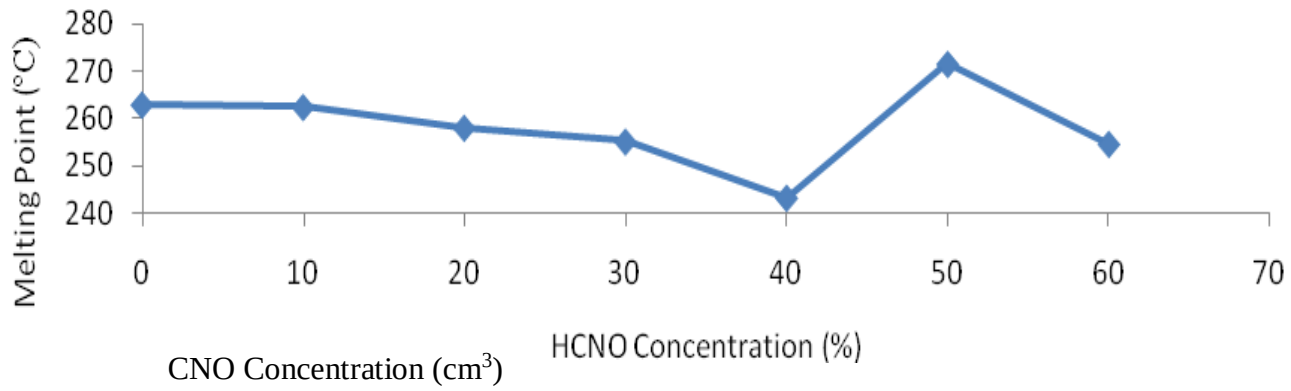

Fig. 4.1.10 Effect of CNO concentration on the melting point of DMU/CNO copolymer composite.

Effect of CNO concentration on the moisture uptake of DMU/CNO copolymer composite.

Figure 4.1.11 shows moisture uptake decreased with CNO loading due to hydrophobicity of CNO (Naghash et al., 2007). This is significant because high moisture uptake is a major drawback of urea-formaldehyde resins, causing blistering and loss of adhesion (Gonzalez et al., 2012). Improved water resistance makes DMU/CNO suitable for waterborne coatings (Emile, 2003).

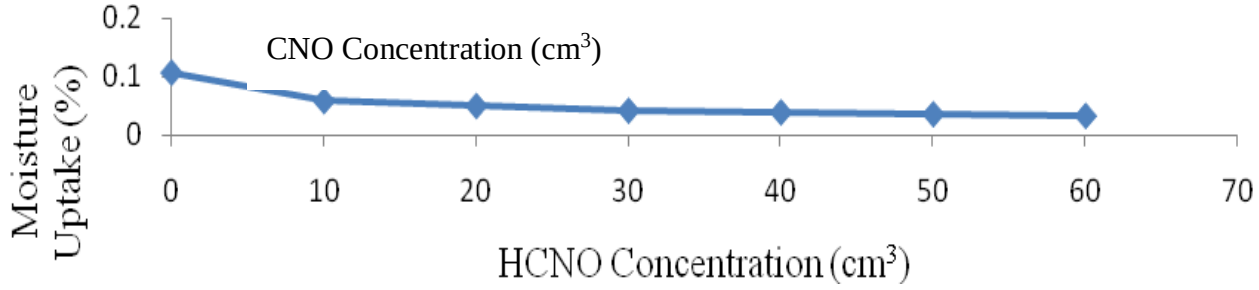

Fig.4.1.11. Effect of CNO concentration on the moisture uptake of DMU/CNO copolymer composite.

Effect of CNO concentration on the elongation at break of DMU/CNO copolymer composite.

Figure 4.1.12 shows elongation at break increased with CNO loading. This is due to CNO providing flexible spacing and increasing free volume (Akinterinwa et al., 2016). Higher elongation indicates reduced brittleness and better film flexibility.

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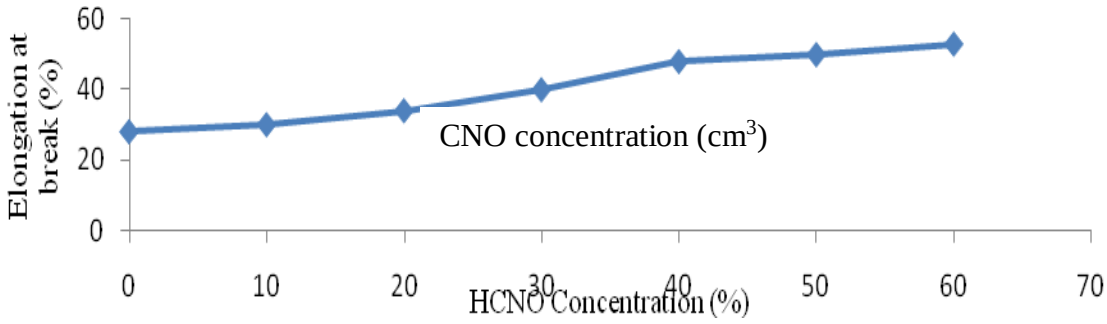


Figure 4.1.12. effect of CNO concentration on the elongation at break of DMU/CNO copolymer composite.

Effect of CNO concentration on the solubility of DMU/CNO copolymer composite.

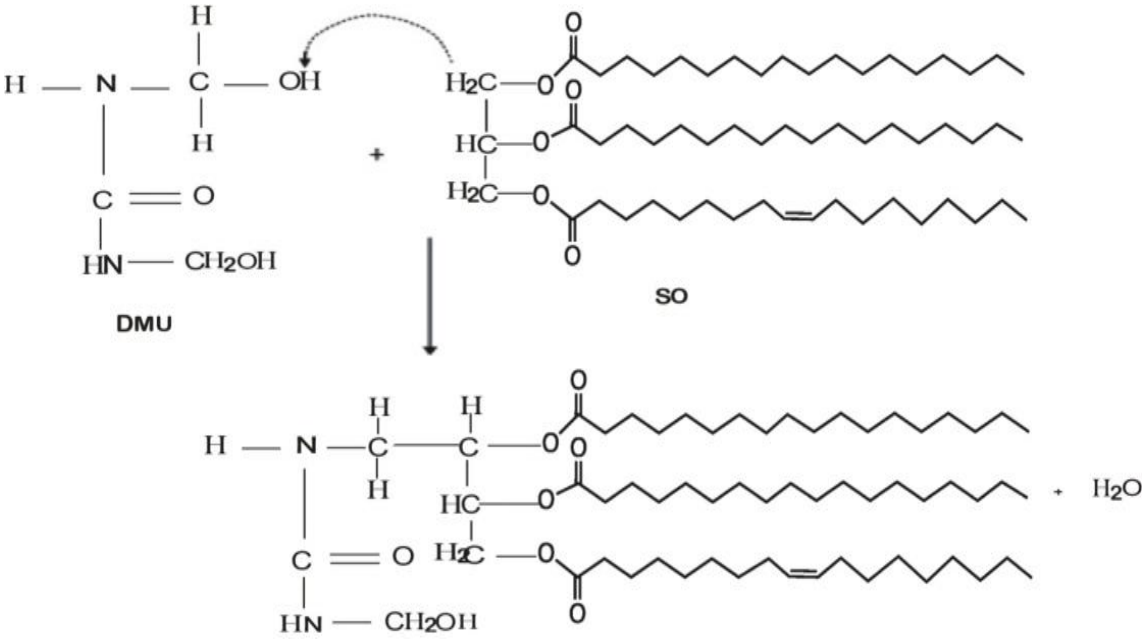
Table 1 shows solubility decreased with CNO loading. The copolymer was soluble up to 40cm³ CNO, then became insoluble. This is because DMU is hydrophilic while CNO is hydrophobic (Osemeahon& Dimas, 2014). For emulsion paint, 30cm³ CNO is the maximum that can stay in aqueous solution.

DMU/CNO concentration	Solubility in water
DMU	Highly soluble
90/10	Soluble
80/20	Soluble
70/30	Slightly soluble
60/40	Insoluble
50/50	Insoluble
40/60	Insoluble

Table 1. Effect of CNO concentration on the solubility of DMU/CNO copolymer composite.

Effect of HSO concentration on some physical properties of DMU/HSO copolymer composite.

Reaction of DMU with SO.



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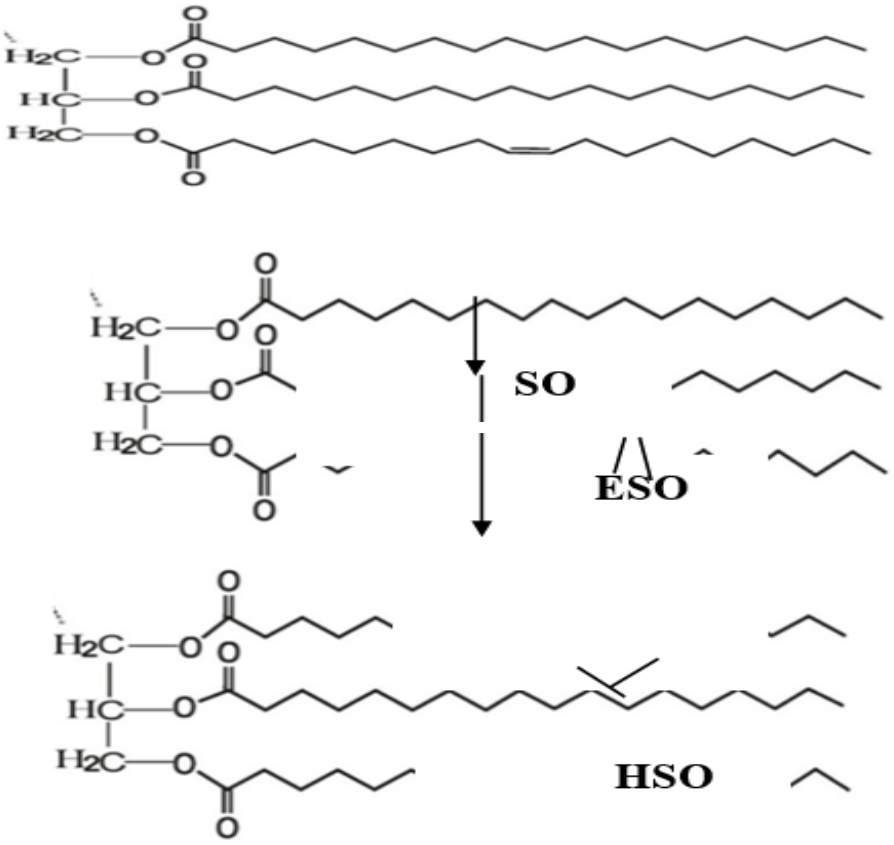
Epoxidation and hydroxylation of SO.



FTIR spectral analysis.

Figures 4.3.3a and 4.3.3b show O-H at 3500-3300 cm^{-1} in HMSO, C=O and C=C in MSO and EMSO. The blend showed O-H at 2933 cm^{-1} and C-O a[illegible] cm^{-1}, confirming interaction (Eric, 2015).

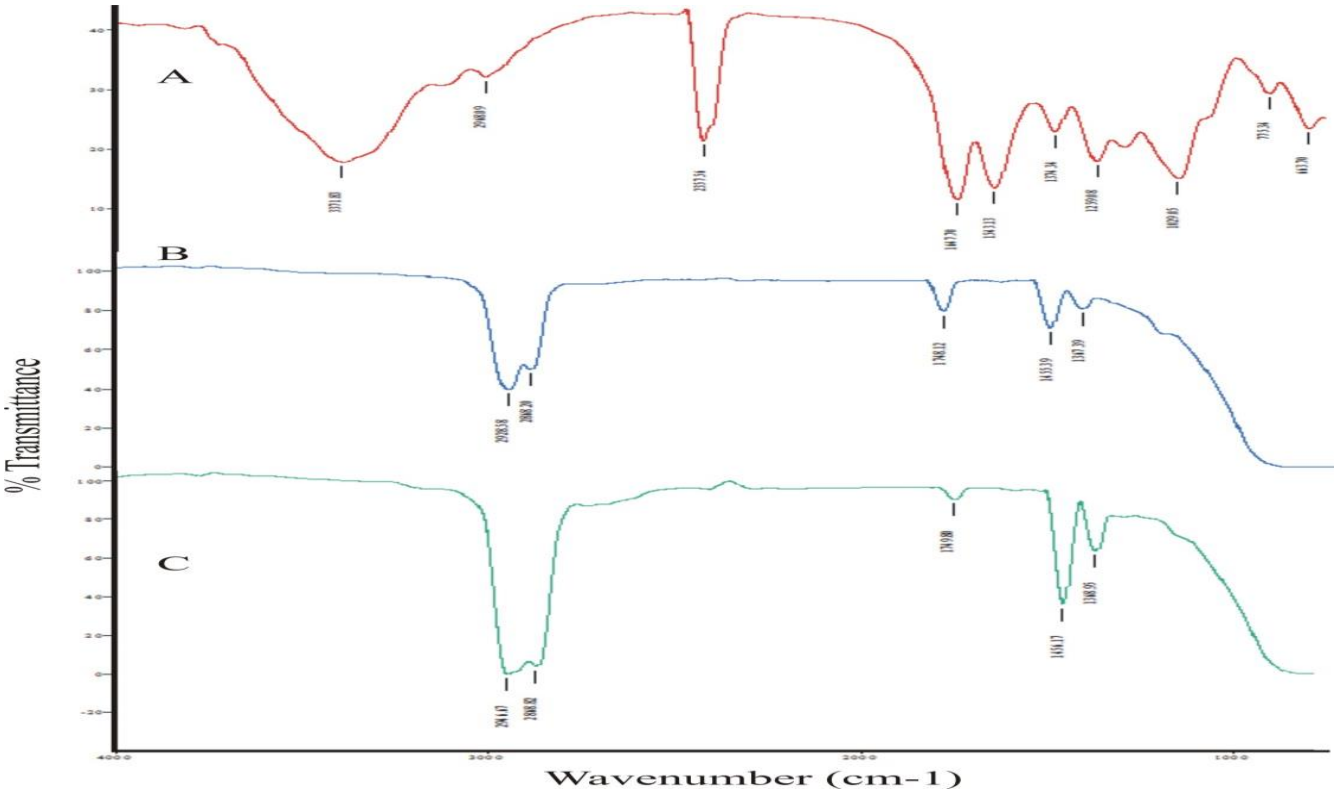


Figure 4.2.3a. A -FTIR spectra of DMU, B- FTIR spectra of SO and C- FTIR spectra of ESO.

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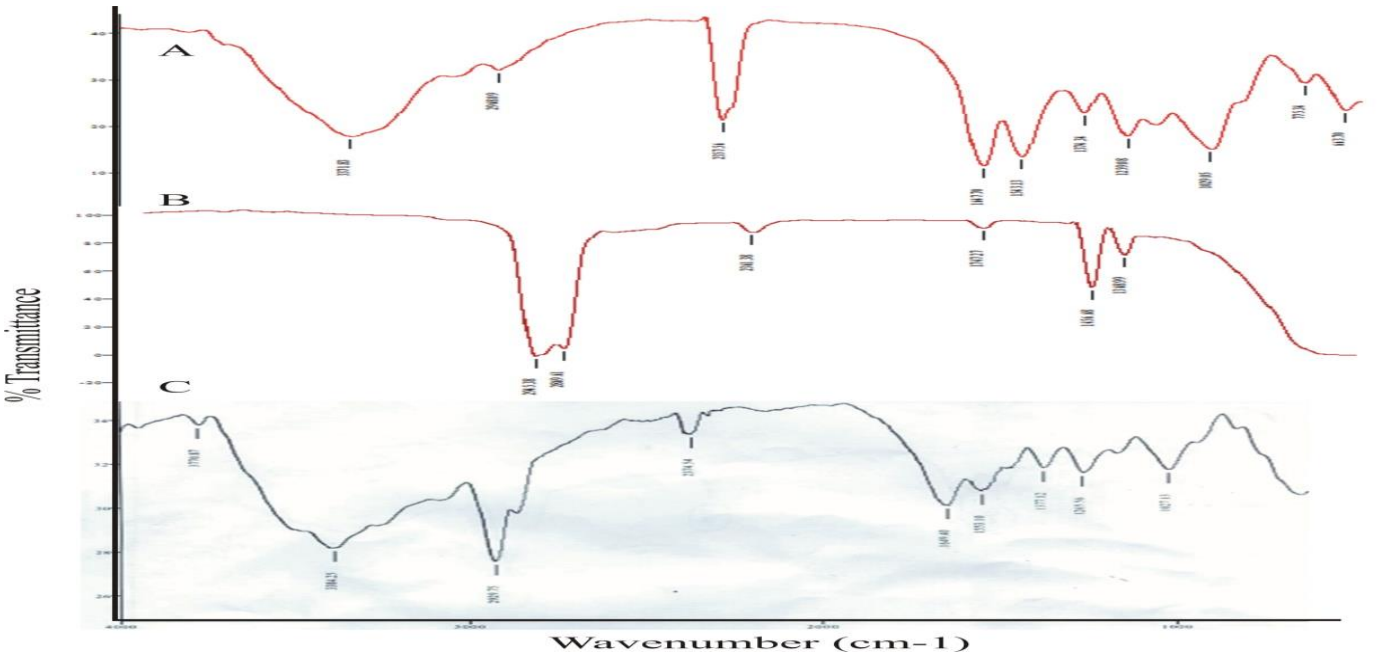

Figure 4.2.3b. A- FTIR spectra of DMU, B- FTIR spectra of HSO and C- FTIR spectra of Blend (DMU/HSO).

Effect of HSO concentration on the viscosity of DMU/HSO copolymer composite.

Figure 4.2.4 shows viscosity increased up to 40cm³ HSO then decreased due to molecular dissociation (Markovic et al., 2001). Viscosity affects leveling, adhesion and drying rate (Osemeahon & Barminas, 2007).

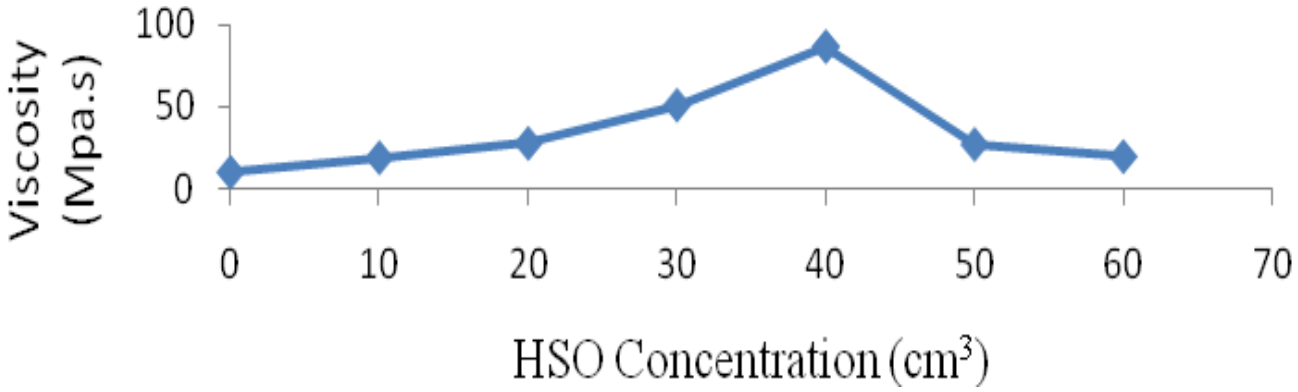

Fig. 4.2.4. Effect of HSO concentration on the viscosity of DMU/HSO copolymer composite.

Effect of HSO concentration on the density of DMU/HSO copolymer composite.

The effect of HSO concentration on the density of DMU/HSO copolymer resin is shown in Figure 4.2.5 shows density decreased with HSO loading due to increase in soft segments (Mavani et al., 2007). Density of blends lies between DMU and HSO (Blaise et al., 2012).

Fig. 4.2.5. Effect of HSO concentration on the density of DMU/HSO copolymer composite.

Effect of HSO concentration on the gel-time of DMU/HSO copolymer composite.

Figure 4.2.6 shows gel-time increased with HSO due to reduced reactivity from bulky alkyl groups (Teware, 2000). Gel-time is important for pot stability and cure rate (Vilas et al., 2000).

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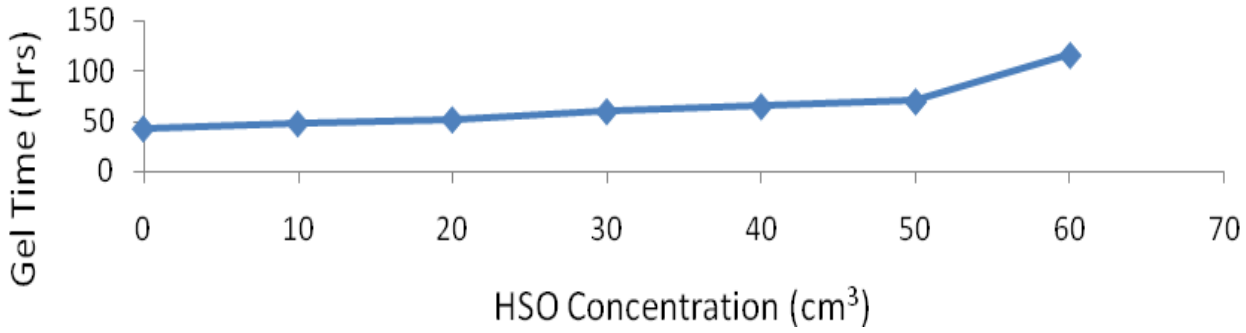

Fig. 4.2.6. Effect of HSO concentration on the gel-time of DMU/HSO copolymer composite.

Effect of HSO concentration on the turbidity of DMU/HSO copolymer composite.

Figure 4.2.7 shows turbidity increased with HSO due to differences in morphology and crystallinity (Johnson & Wilkes, 2001). This affects gloss of the paint film (Trezza & Krochta, 2001).

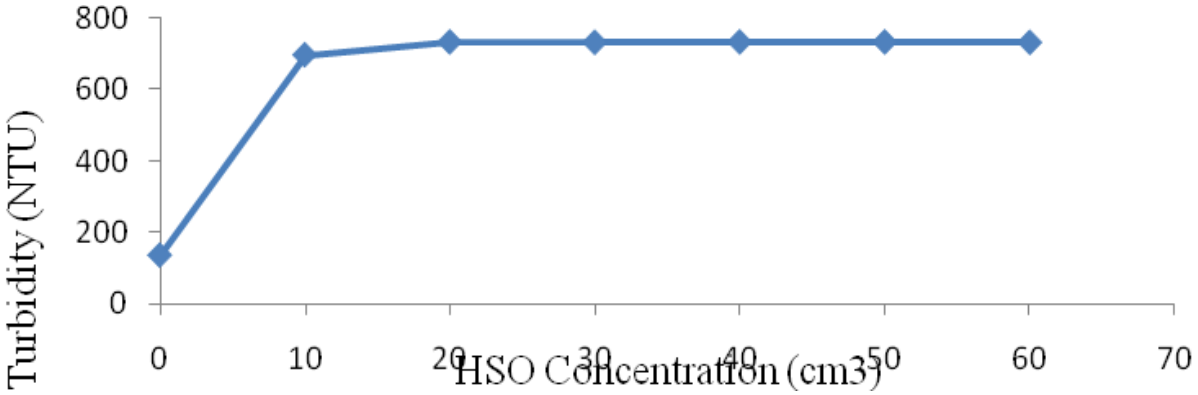

Fig. 4.2.7. Effect of HSO concentration on the turbidity of DMU/HSO copolymer composite.

Effect of HSO concentration on the refractive index of DMU/HSO copolymer composite.

Figure 4.2.8 shows refractive index increased with HSO due to higher molecular weight (Trezza & Krochta, 2001). This improves gloss properties.

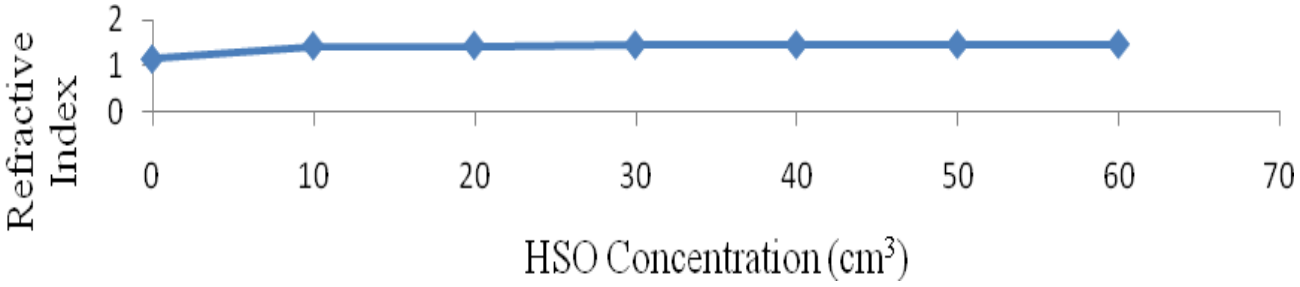

Fig. 4.2.8. Effect of HSO concentration on the refractive index of DMU/HSO copolymer composite.

Effect of HSO concentration on the formaldehyde emission of DMU/HSO copolymer composite.

Figure 4.2.9 shows emission decreased with HSO loading due to dilution of DMU and disruption of DMU matrix (Pizzi et al., 2001; Lee et al., 2011). This addresses a major drawback of urea-formaldehyde resins (Kim, 2001).

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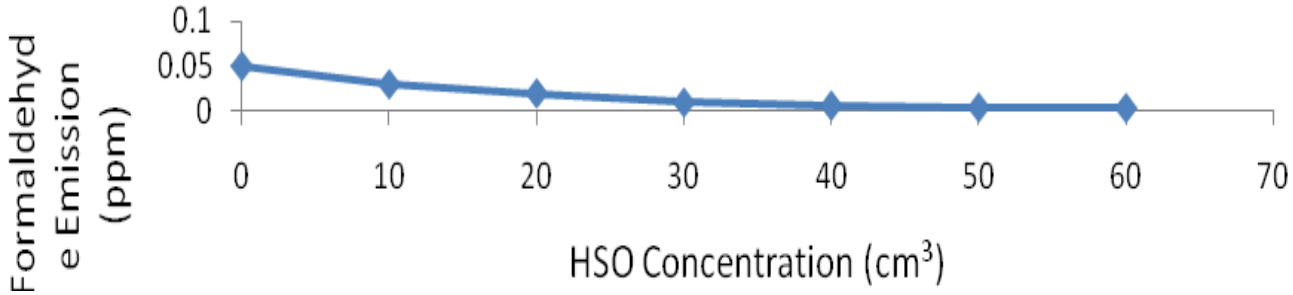

Fig. 4.2.9. Effect of HSO concentration on the formaldehyde emission of DMU/HSO copolymer composite

Effect of HSO concentration on the melting point of DMU/HSO copolymer composite.

Figure 4.2.10 shows melting point decreased then increased at 50cm³ due to increased molecular weight and association. Melting point relates to thermal resistance and rigidity (Park et al., 2002).

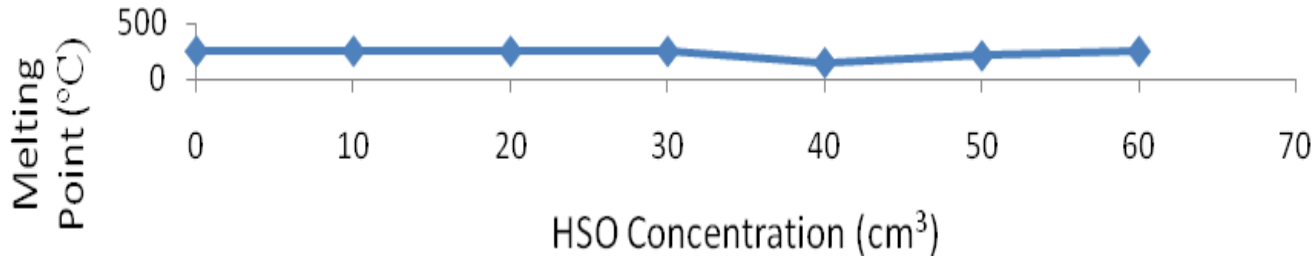

Fig. 4.2.10. Effect of HSO concentration on the melting point of DMU/HSO copolymer composite.

Effect of HSO concentration on the elongation at break of DMU/HSO copolymer composite.

Figure 4.2.11 shows elongation increased with HSO due to more soft segments and flexibility (Akinterinwa et al., 2015). This overcomes brittleness of DMU (Osemeahon & Barminas, 2007).

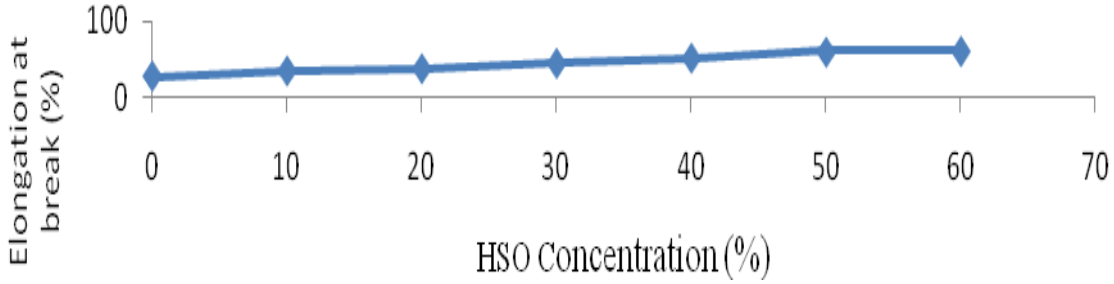

Figure 4.2.11. Effect of HSO concentration on the elongation at break of DMU/HSO copolymer composite.

Effect of HSO concentration on the moisture uptake of DMU/HSO copolymer composite.

Figure 4.2.12 shows moisture uptake decreased with HSO due to hydrophobicity and reduced polymer-water affinity (Nogueira et al., 2001). This improves water resistance of DMU.

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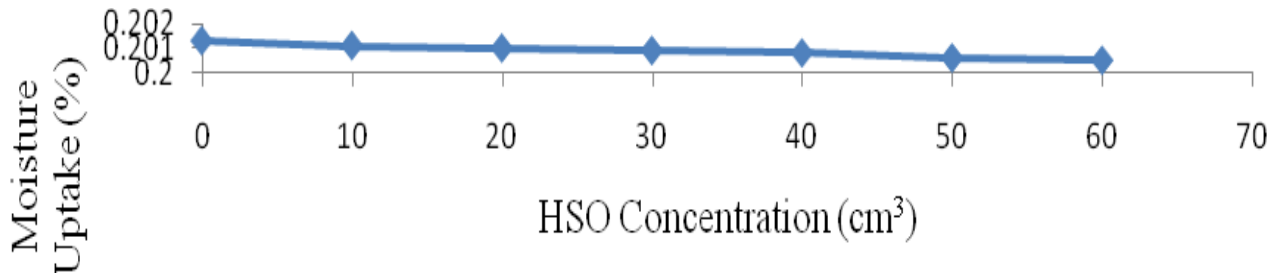


Fig. 4.2.12. Effect of HSO concentration on the moisture uptake of DMU/HSO copolymer composite.

Effect of HSO concentration on solubility of DMU/HSO copolymer composite.

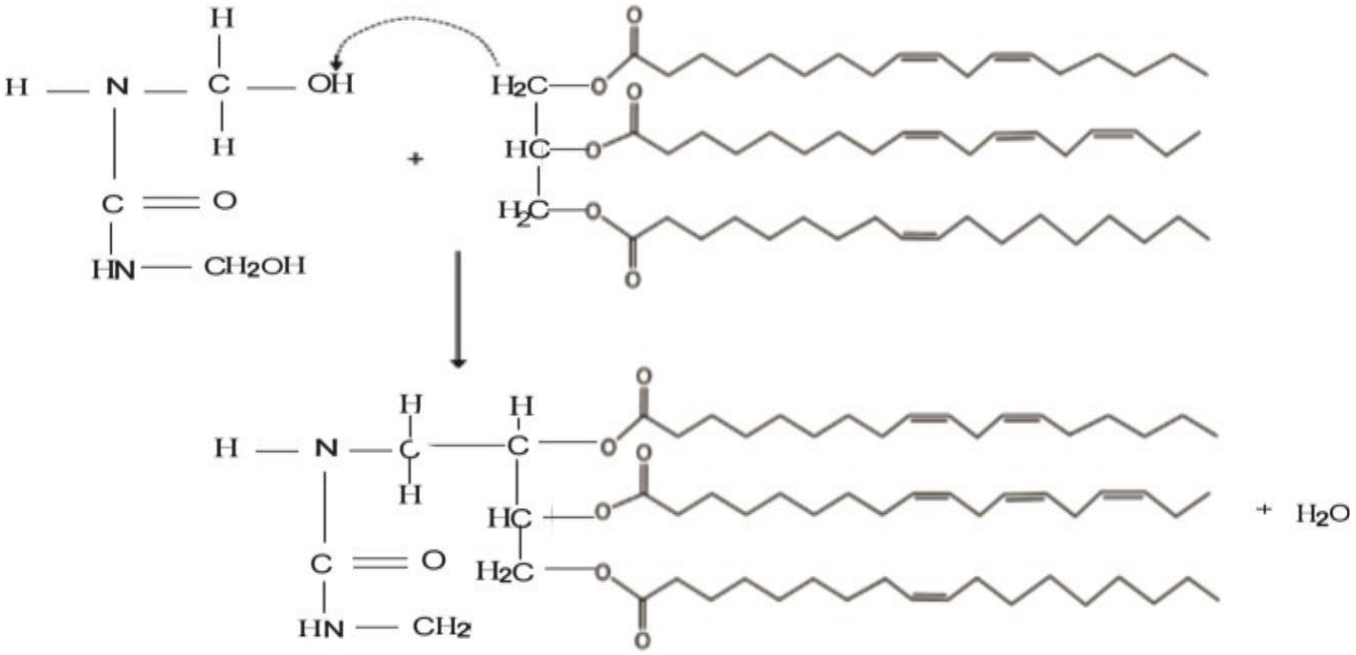
Table 2 shows copolymer was soluble up to 40cm³ HSO, then insoluble. Water solubility is important for emulsion paint binders.

DMU/HSO concentration	Solubility in water
0	Highly soluble
90/10	Soluble
80/20	Soluble
70/30	Slightly soluble
60/40	Slightly soluble
50/50	Insoluble
40/60	Insoluble

Table 2. Effect of HSO concentration on the solubility of DMU/HSO copolymer composite.

Effect of HMSO concentration on some physical properties of DMU/HMSO copolymer composite.

Reaction of DMU with MSO



FTIR spectral analysis.

Figures 4.3.3a and 4.3.3b show O-H at 3500-3300 cm^{-1} in HMSO, C=O and C=C in MSO and EMSO. The blend showed O-H at 2933 cm^{-1} and C-O at 1261 cm^{-1}, confirming interaction (Eric, 2015).

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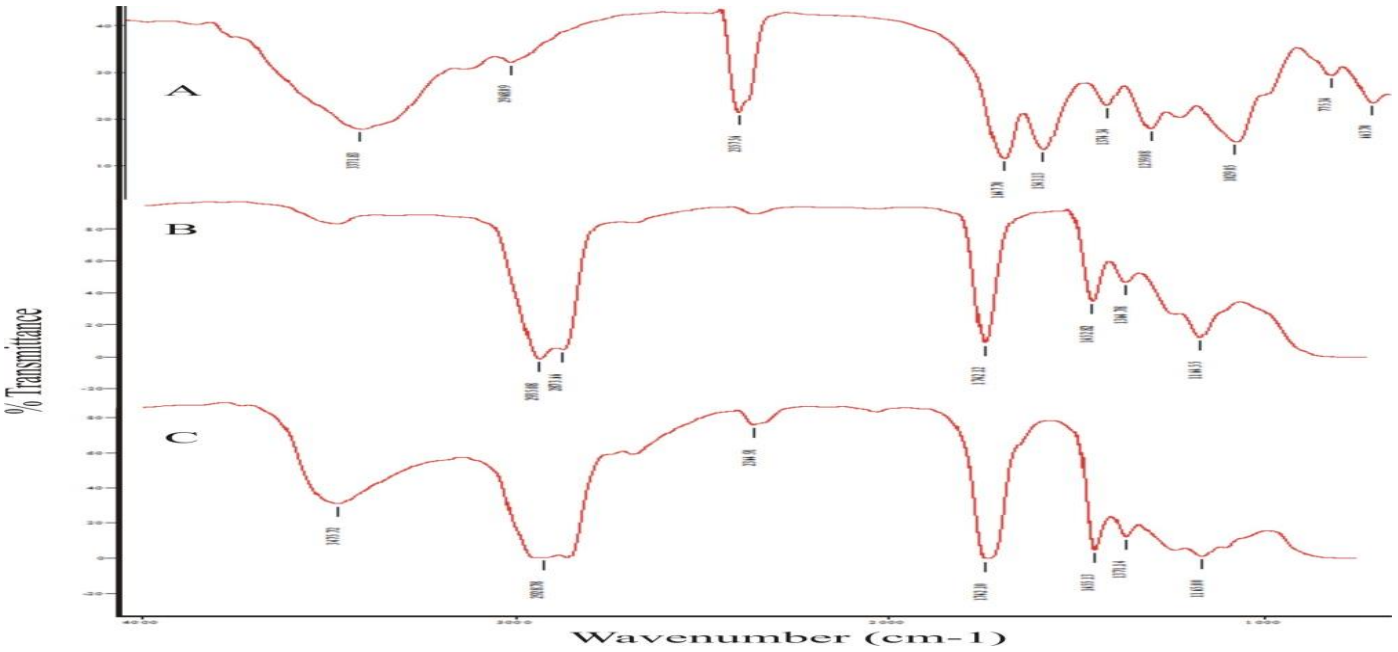

Figure 4.3.2 A- FTIR spectra of DMU, B- FTIR spectra of MSO and C- FTIR spectra of EMSO.

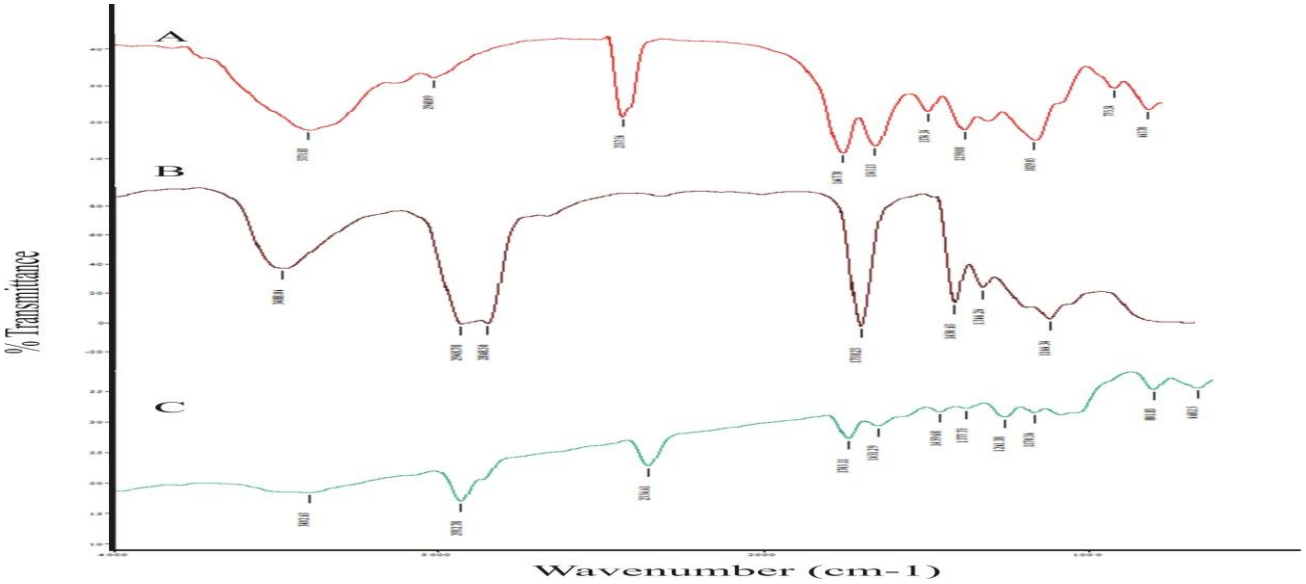

Figure 4.3.3 A- FTIR spectra of DMU, B- FTIR spectra of HMSO and C- FTIR spectra of Blend (DMU/HMSO).

Effect of HMSO concentration on the viscosity of DMU/HMSO copolymer composite.

Figure 4.3.4 shows viscosity peaked at 50cm³ HMSO. Up to 50cm³, hard DMU segments formed continuous phase. Beyond that, soft HMSO formed continuous phase, reducing viscosity (Qi et al., 2002). Viscosity controls flow and adhesion (Osemeahon & Barminas, 2007).

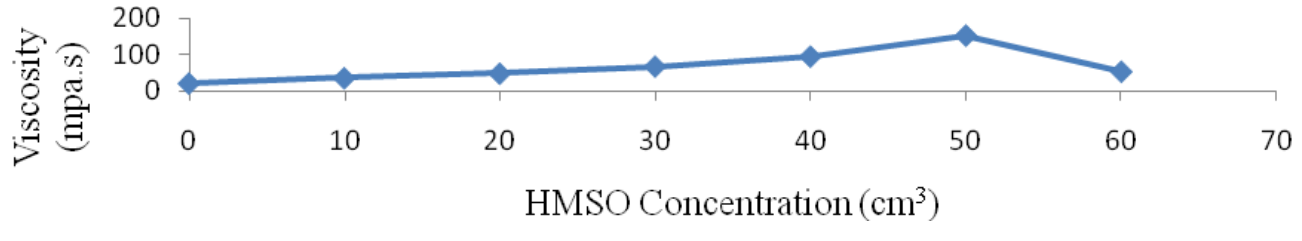

Fig. 4.3.4. Effect of HMSO concentration on the viscosity of DMU/HMSO copolymer comosite.

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Effect of HMSO concentration on the density of DMU/HMSO copolymer composite.

Figure 4.3.5 shows density decreased with HMSO due to poor chain packing and increase in soft segments (Osemeahon & Archibong, 2011).

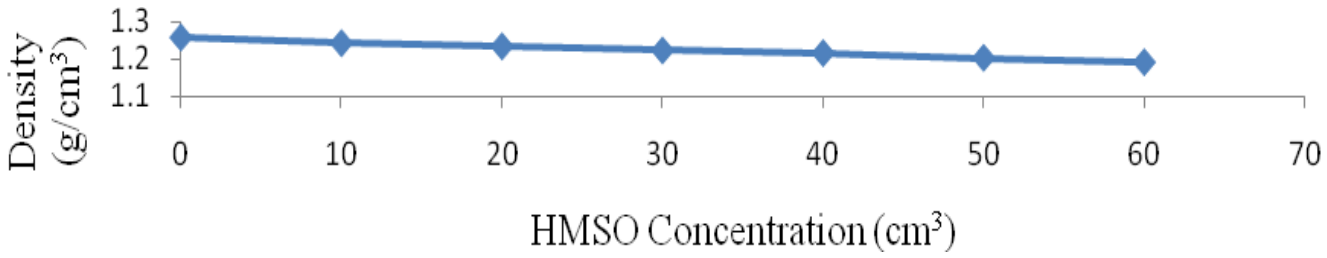

Fig. 4.3.5. Effect of HMSO concentration on the density of DMU/HMSO copolymer composite.

Effect of HMSO concentration on the gel-time of DMU/HMSO copolymer composite.

Figure 4.3.6 shows gel-time increased with HMSO due to reduced reactivity (Osemeahon, 2011). This affects drying and storage.

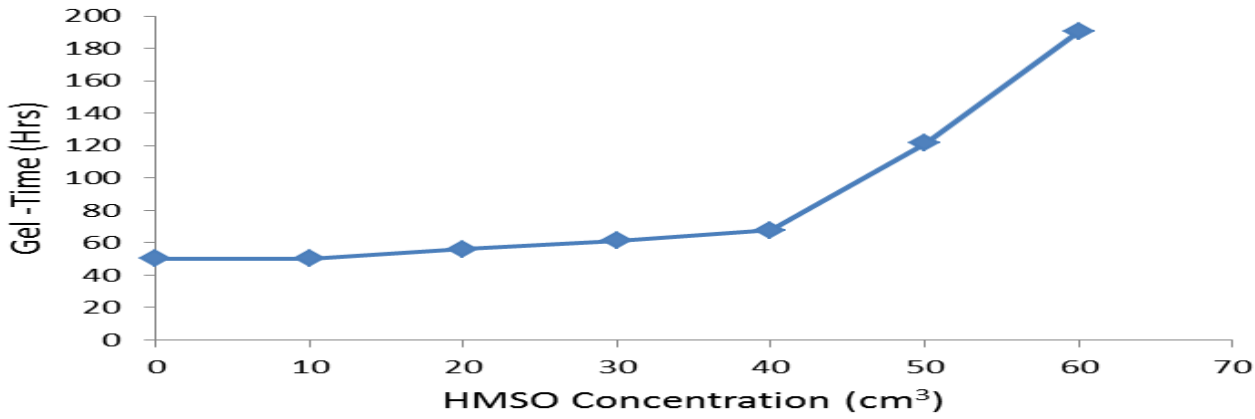

Fig. 4.3.6. Effect of HMSO concentration on the gel-time of DMU/HMSO copolymer composite.

Effect of HMSO concentration on the turbidity of DMU/HMSO copolymer composite.

Figure 4.3.7 shows turbidity increased with HMSO due to light scattering from changes in crystallinity (Moreno et al., 2000).

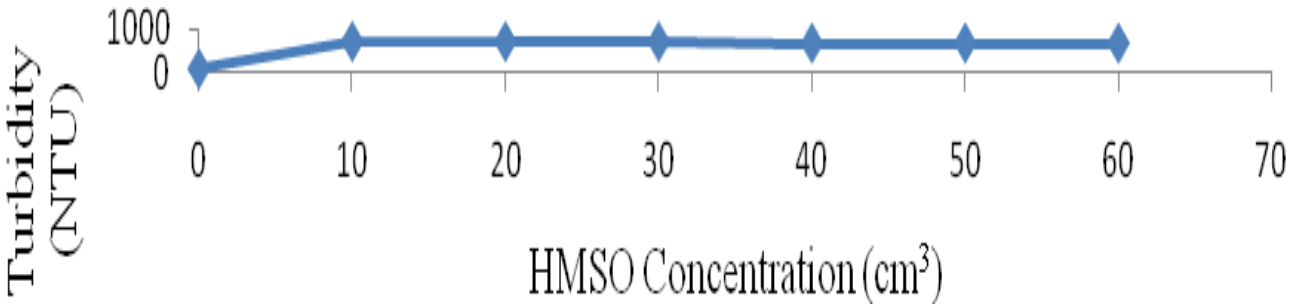

Fig. 4.3.7. Effect of HMSO concentration on the turbidity of DMU/HMSO copolymer composite.

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Effect of HMSO concentration on the refractive index of DMU/HMSO copolymer composite.

Figure 4.3.8 shows refractive index increased with HMSO due to changes in molecular structure and crosslinking (Qi et al., 2002).

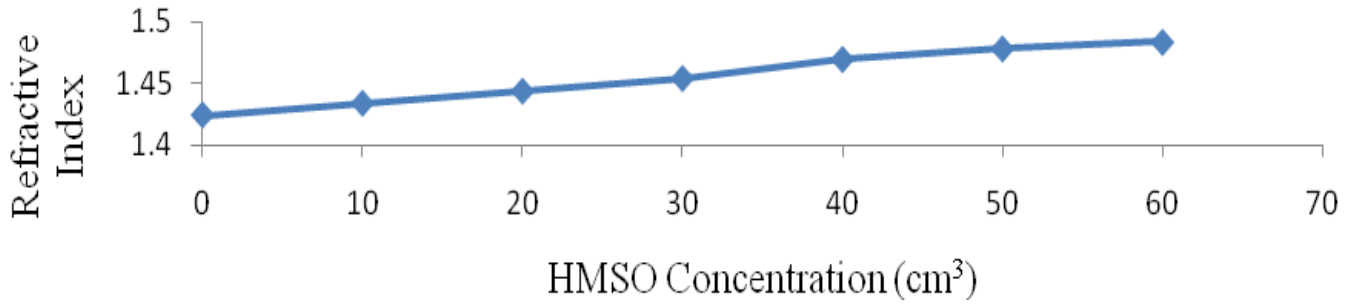

Fig. 4.3.8. Effect of HMSO concentration on the refractive index of DMU/HMSO copolymer composite.

Effect of HMSO concentration on the formaldehyde emission of DMU/HMSO copolymer composite.

Figure 4.3.9 shows emission decreased with HMSO due to reduced DMU and interaction hindering condensation (Akinterinwa et al., 2015).

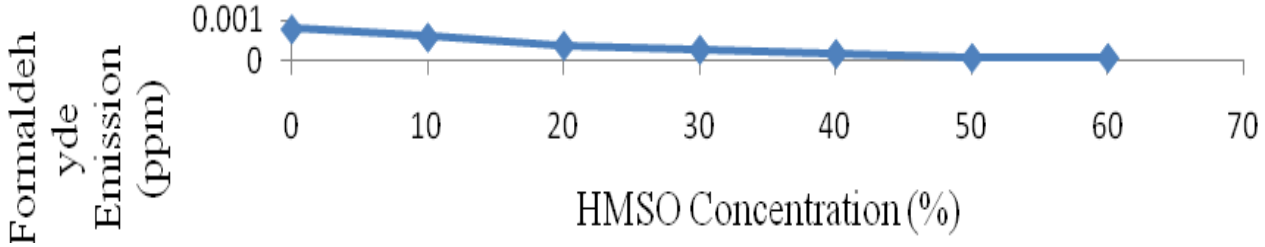

Fig. 4.3.9. Effect of HMSO concentration on the formaldehyde emission of DMU/HMSO copolymer composite.

Effect of HMSO concentration on the melting point of DMU/HMSO copolymer composite.

Figure 4.3.10 shows melting point decreased, became steady, then increased due to polymer association (Osemeahon & Dimas, 2014).

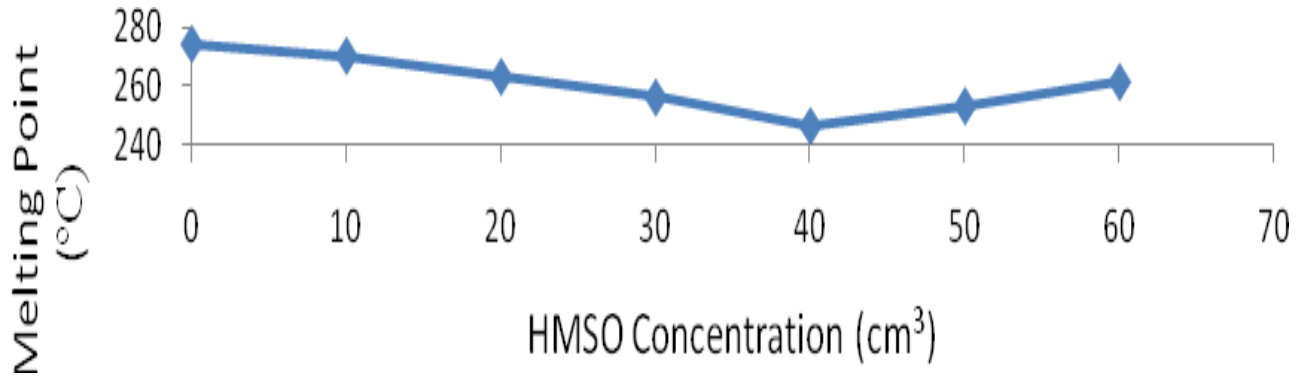

Fig. 4.3.10. Effect of HMSO concentration on the melting point of DMU/HMSO copolymer composite.

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Effect of HMSO concentration on the moisture uptake of DMU/HMSO copolymer composite.

Figure 4.3.11 shows moisture uptake decreased with HMSO due to hydrophobicity (Akinterinwa et al., 2015).

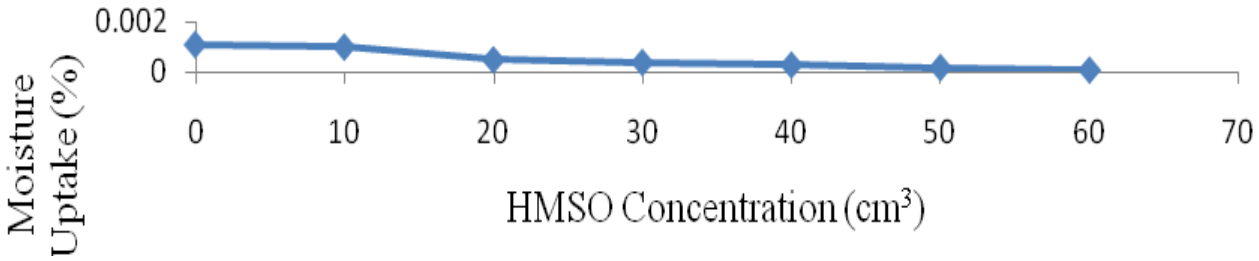

Fig. 4.3.11. Effect of HMSO concentration on the moisture uptake of DMU/HMSO copolymer composite.

Effect of HMSO concentration on the elongation at break of DMU/HMSO copolymer composite.

Elongation at break is a significant factor recounting the rupture performance of composites. It is the ratio between increased length and initial length after breakage of a tested material (Wu, *et al*., 2000).

The effect of HMSO concentration on the elongation at break of DMU/HMSO copolymer composite is shown in Figure 4.3.12 shows elongation increased with HMSO due to increased flexibility.

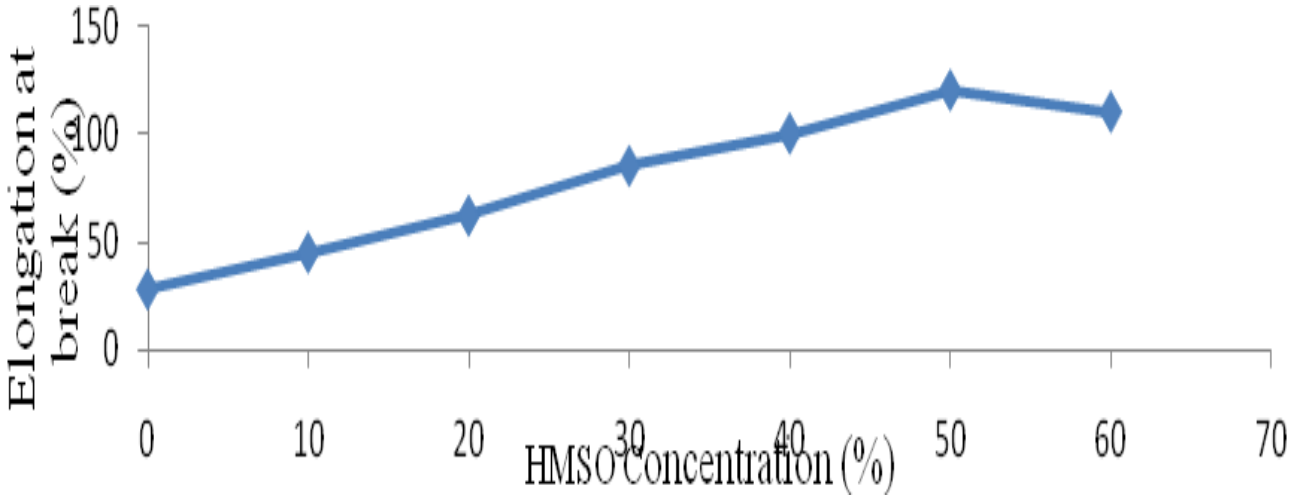

Figure 4.3.12. Effect of HMSO concentration on the elongation at break of DMU/HMSO copolymer composite.

Effect of HMSO concentration on the solubility of DMU/HMSO copolymer composite.

Table 3 shows solubility up to 40cm³, then insolubility due to hydrophobic HMSO (Akinterinwa et al., 2016).

DMU/HMSO concentration	Solubility in water
0	Highly soluble
90/10	Soluble
80/20	Soluble
70/30	Soluble
60/40	Slightly soluble
50/50	Insoluble
40/60	Insoluble

Table 3. Effect of HMSO concentration on the solubility of DMU/HMSO copolymer composite.

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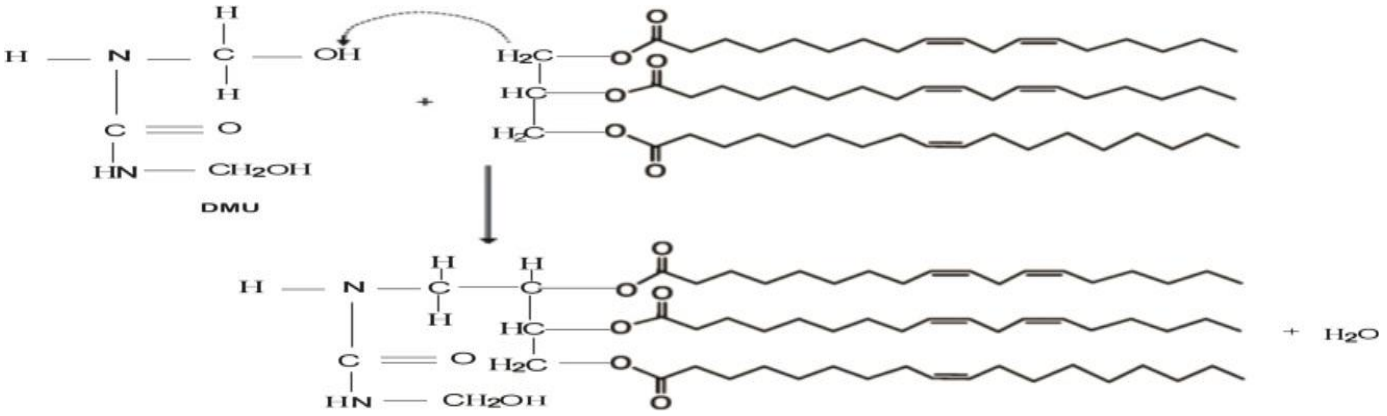
Effect of HBSO concentration on some physical properties of DMU/HBSO copolymer composite.

Reaction of DMU with BSO.



Epoxidation and hydroxylation of BSO.

FTIR spectral analysis

Similar trends were observed for HBSO: FTIR confirmed copolymerization; viscosity, gel-time, turbidity and refractive index increased to an optimum then decreased; density, melting point and moisture uptake decreased; elongation increased; solubility decreased with loading (Tables and Figures 4.4.3 – 4.4.13). The hydrophobic nature of HBSO improved water resistance while reducing formaldehyde emission (Osemeahon, 2011).

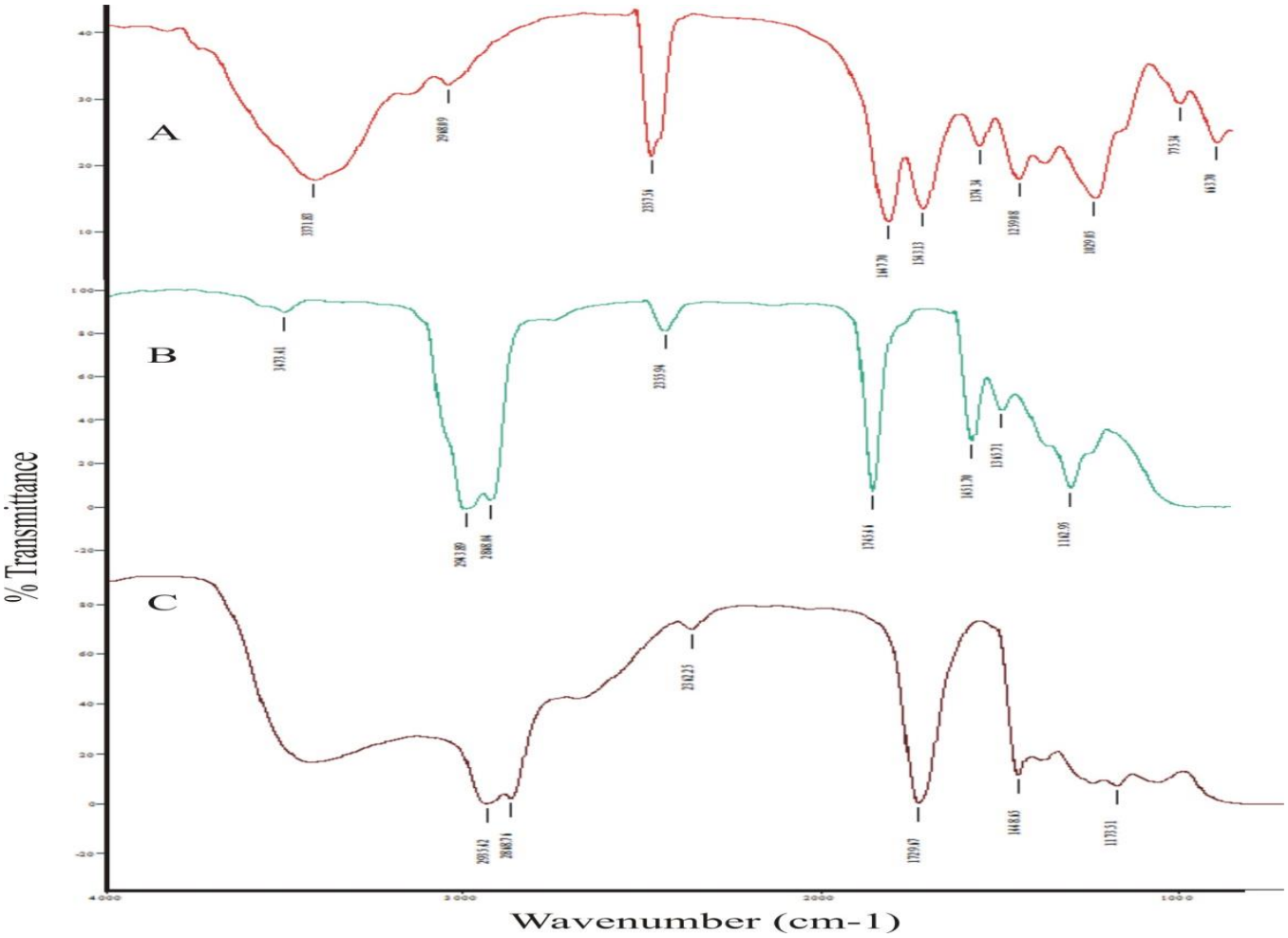


Figure 4.4.3a

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A- FTIR spectra of DMU, B- FTIR spectra of BSO and C- FTIR spectra of EBSO.

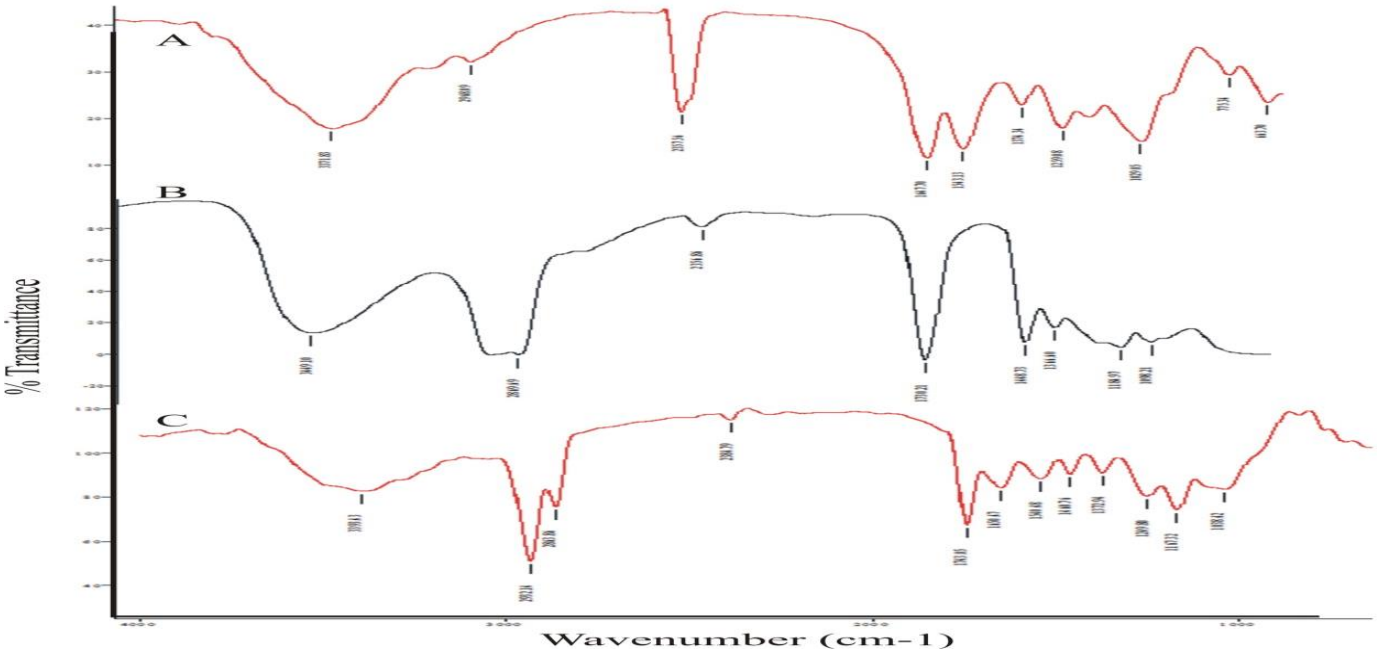

Figure 4.4.3b

A- FTIR spectra of DMU, B- FTIR spectra of HBSO and C- FTIR spectra of Blend (DMU/HBSO).

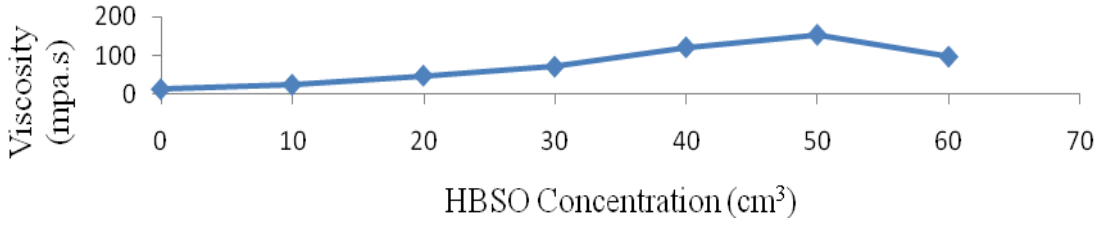

Fig. 4.4.4. Effect of HBSO concentration on the viscosity of DMU/HBSO copolymer composite.

Effect of HBSO concentration on the density of DMU/HBSO copolymer composite.

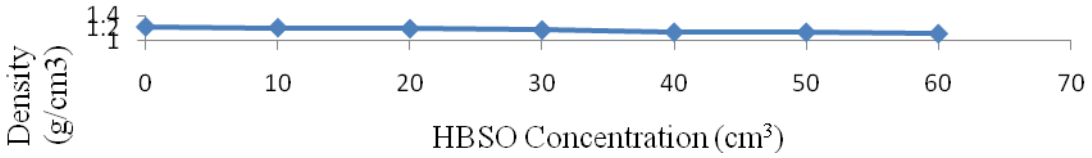

Fig. 4.4.5. Effect of HBSO concentration on the density of DMU/HBSO copolymer composite.

Effect of HBSO concentration on the gel-time of DMU/HBSO copolymer composite.

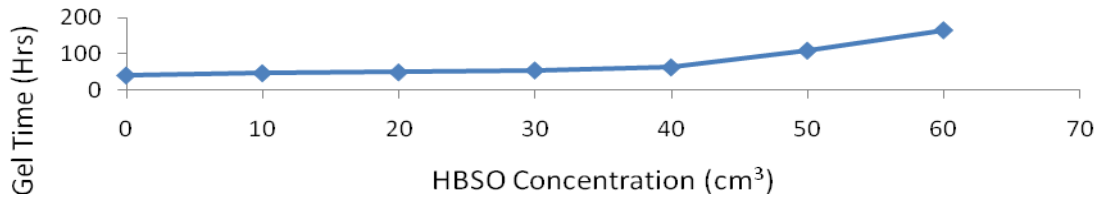

Fig. 4.4.6. Effect of HBSO concentration on the gel-time of DMU/HBSO copolymer composite.

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Effect of HBSO concentration on the turbidity of DMU/HBSO copolymer composite.

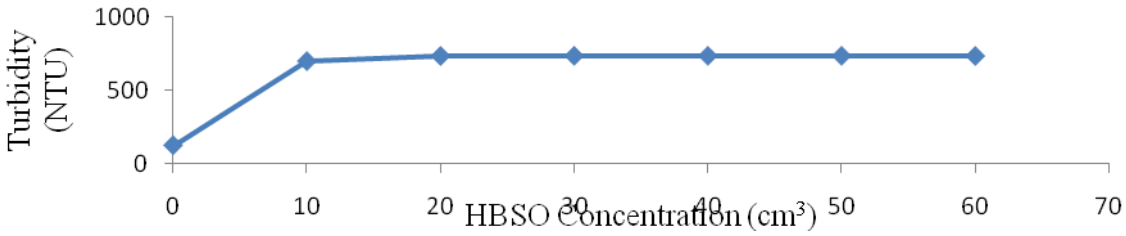

Fig. 4.4.7. Effect of HBSO concentration on the turbidity of DMU/HBSO copolymer composite.

Effect of HBSO concentration on the refractive index of DMU/HBSO copolymer composite.

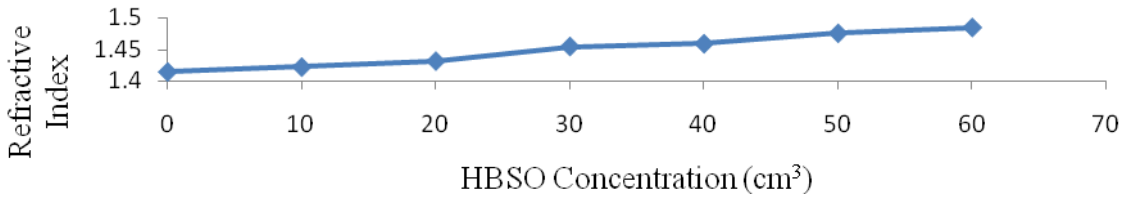

Fig. 4.4.8. Effect of HBSO concentration on the refractive index of DMU/HBSO copolymer composite.

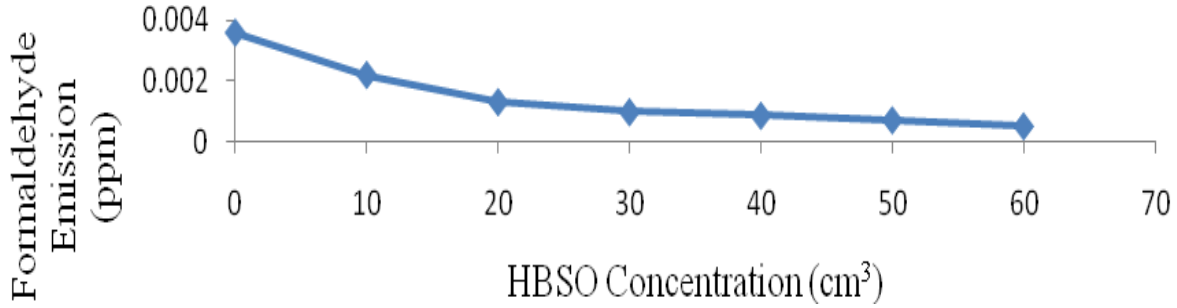

Fig. 4.4.9. Effect of HBSO concentration on the formaldehyde emission of DMU/HBSO copolymer composite.

Effect of HBSO concentration on the melting point of DMU/HBSO copolymer composite.

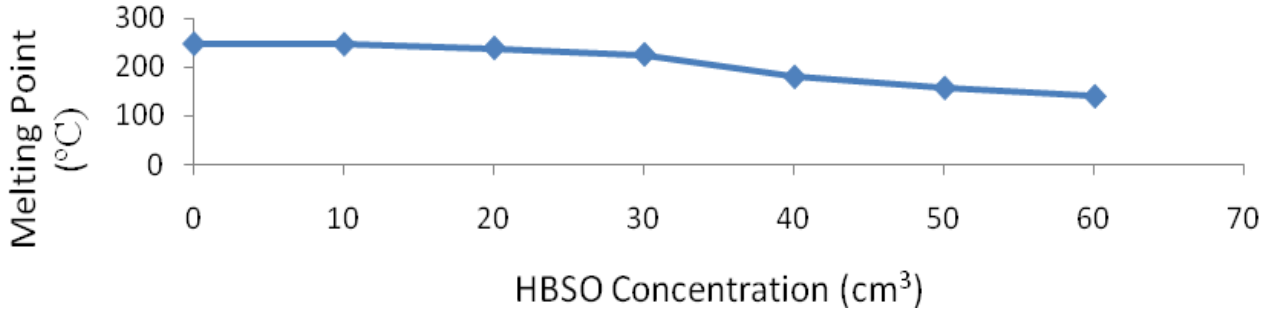

Fig. 4.4.10. Effect of HBSO concentration on the melting point of DMU/HBSO copolymer composite.

Effect of HBSO concentration on the moisture uptake of DMU/HBSO copolymer composite.

Fig. 4.4.11. Effect of HBSO concentration on the moisture uptake of DMU/HBSO copolymer composite.

Effect of HBSO concentration on the elongation at break of DMU/HBSO copolymer composite.

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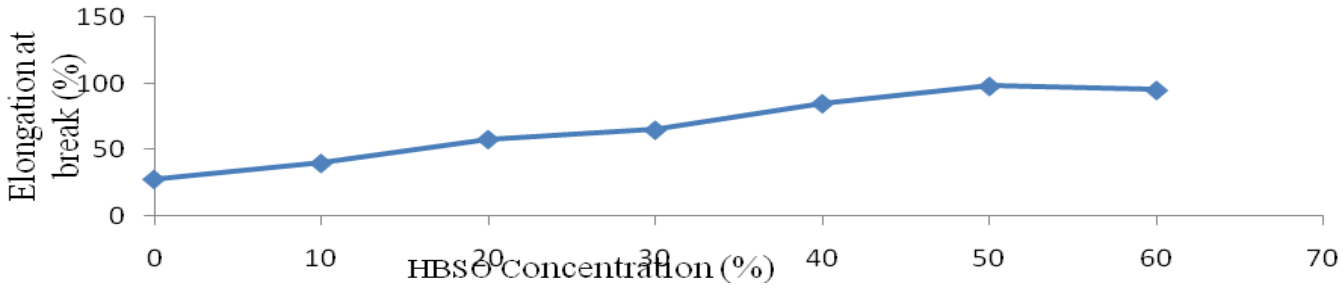

Figure 4.4.12. Effect of HBSO concentration on the elongation at break of DMU/HBSO copolymer composite.

Effect HBSO concentration on the solubility of DMU/HBSO copolymer composite.

DMU/HBSO concentration	Solubility in water
0	Highly soluble
90/10	Soluble
80/20	Soluble
70/30	Soluble
60/40	Slightly soluble
50/50	Insoluble
40/60	Insoluble

Table 4. Effect of HBSO concentration on the solubility of DMU/HBSO copolymer composite.

Analysis of some physical properties of paint using DMU/HVO's copolymer as binder.

Some physical properties of EP using pure DMU, PVA, DMU/HCNO, DMU/HSO, DMU/HMSO, and DMU/HBSO as binders were analysed. The properties were compared to that of pure EP (PVA was used as binder) and analysed as well. The results are tabulated in tables 5a, 5b, 5c and 5d, and a general comparison on table 6 respectively.

Table 5a shows some physical properties of EP formulated with pure DMU, PVA and DMU/HCNO as binders. The viscosity of the EP formulated from DMU/HCNO was observed to be higher than that of DMU, and the standard value respectively. This is as a result of copolymerization between DMU and the HCNO. This result is expected because of the increase in molecular weight of the copolymer that is formed as increase in molecular weight gave rise to increase in viscosity (Afzal *et al*., 2013). It is also observed that the pH is slightly above the standard value. This is a fair result as it does not go too far beyond the standard range.

The paint from DMU/HCNO copolymer passed the test for properties such as opacity, drying time, flexibility, adhesion, tackiness, resistance to blistering, stability and chemical resistance. This is a plus to the EP/DMU/HCNO formulation as the integrity of the important factors is maintained. The gloss of EP from DMU/HCNO formulation is better than that of the DMU EP. This result is expected because CNO has a higher refractive index than DMU. This also is seen as an added advantage to the development of a new paint binder using DMU/HCNO as a binder for the formulation of EP.

Table 5a: Some physical properties of paint formulated from pure DMU, PVA and DMU/HCNO as binders.

S/no	Parameter	DMU	DMU/HCNO	PVA	Standard (SON)
1	Viscosity (poise)	42.64	45.22	7.5	6 – 15
2	pH	8.62	8.77	8.80	7 – 8.5
3	Opacity	G	G	V.G	P
4	Drying time(min)	TD 15	16	17	20
		HD 87	89	75	120

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5	Flexibility	P	P	P	P
6	Adhesion	V.G	G	G	P
7	Tackiness	P	P	P	P
8	Resistance to blistering	P	P	P	P
9	Stability	S	S	S	P
10	Chemical resistance	P	P	P	P
11	Gloss @85^{O}	20	22	18	16 – 50

P: Pass, TD: Touch dry, V.G: Very good, G: Good, S: Stable, HD: Hard dry

Table 5b shows some physical properties of EP formulated using pure DMU, PVA and DMU/HSO as binders. From the table, it is observed that the viscosity of EP formulated using DMU/HSO as binder is higher than those from the pure DMU and the standard values. This result may be attributed to the variation of the different chain lengths present in the copolymer, as DMU copolymerizes with HSO (Markovic, *et al.*, 2001). It is also observed that the pH value for this formulation is slightly above that of the standard value. It is a fair result as it does not go too far beyond the standard range. The gloss of EP formulated from DMU/HSO is higher than that of the pure EP formulated with PVA as binder. This is an expected result because the refractive index of SO is high. It is an advantage to the development of a new paint binder using DMU/HSO for the formulation of EP. The EP formulated using DMU/HSO as binder passed the test for various properties such as opacity, drying time, flexibility, adhesion, tackiness, resistance to blistering, stability and chemical resistance. This also is a plus to the DMU/HSO formulation because the integrity of the important factors is maintained.

Table 5b: Some physical properties of paint formulated from pure DMU, PVA and DMU/HSO as binders.

S/no	Parameter	DMU	DMU/HSO	PVA	Standard(SON)
1	Viscosity(poise)	42.64	45.22	7.5	6 – 15
2	pH	8.62	8.77	8.80	7 – 8.5
3	Opacity	G	G	V.G	P
4	Drying time(min)	TD 15	19	17	20
		HD 87	89	75	120
5	Flexibility	P	P	P	P
6	Adhesion	V.G	G	G	P
7	Tackiness	P	P	P	P
8	Resistance to blistering	P	P	P	P
9	Stability	S	S	S	P
10	Chemical resistance	P	P	P	P
11	Gloss @ 85^{O}	20	20	18	16 – 50

P: Pass, TD: Touch dry, S: Stable, HD: Hard dry, V.G: Very good, G: Good

Table 5c shows some physical properties of EP formulated using DMU, PVA and DMU/HMSO as binders. The viscosity of EP formulated using DMU/HMSO as binder is higher than that of the pure EP in which PVA was used as a binder, and that of the standard values as can be observed from the table. This result may be attributed to the segmental factor of the resin. With the DMU alone as the binder, it is observed that the viscosity is lower than the copolymer due to lower molecular weight. However, as HMSO, which is the soft segment is introduced, there is a strong association between the DMU and HMSO forming large aggregates with large hydrodynamic volumes which lead to high viscosity. The pH for this formulation is also observed to be slightly above that of the standard value. This is also a fair result as it does not go too far beyond the standard range. The gloss of EP formulated from DMU/HMSO as binder is higher than that of the pure EP which was formulated from PVA. This also is an expected result as the refractive index of MSO is high. This is an advantage to the development of a new paint binder using DMU/HMSO for the formulation of EP. The

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EP formulated from DMU/HMSO passed the test for various properties such as opacity, drying time, flexibility, adhesion, tackiness, resistance to blistering, stability and chemical resistance. This is a plus to the EP/ from DMU/HMSO formulation because the properties of the important factors here are also maintained.

Table 5c: Some physical properties of paint formulated from pure DMU, PVA and DMU/HMSO as binders.

S/no	Parameter	DMU	DMU/HMSO	PVA	Standard(SON)
1	Viscosity(poise)	42.64	45.22	7.5	6 – 15
2	pH	8.62	8.77	8.80	7 – 8.5
3	Opacity	G	G	V.G	P
4	Drying	TD 15	17	17	20
	time(min)	HD 87	85	75	120
5	Flexibility	P	P	P	P
6	Adhesion	V.G	G	G	P
7	Tackiness	P	P	P	P
8	Resistance to blistering	P	P	P	P
9	Stability	S	S	S	P
10	Chemical resistance	P	P	P	P
11	Gloss@ 85^{O}	20	19	18	16 - 50

P: Pass, TD: Touch dry, S: Stable, HD: Hard dry, V.G: Very good, G: Good

Table 5d shows some physical properties of EP formulated using pure DMU, PVA and DMU/HBSO as binders. The viscosity of EP formulated using DMU as binder is higher than that of the EP formulated from PVA, and that of the standard value as can be observed from the table. This result may be attributed to the differentials in the molecular weight and crosslinked density among the different amino resins. As the alkyl chains increases, the molecular weight also increases thereby increasing the viscosity (Markovic *et al.*, 2001). The gloss of EP from DMU/HBSO formulation is observed to be the same as that of the pure EP from PVA. This is an advantage to the development of a new paint binder using DMU/HBSO for the formulation of EP, as most of the properties meet that of the standard range. The EP from DMU/HBSO passed the test for various properties such as opacity, drying time, flexibility, adhesion, tackiness, resistance to blistering, stability, chemical resistance, pH and gloss. This is a plus to the DMU/HBSO formulation because the integrity of the important factors here is also maintained.

Table 5d: Some physical properties of paint formulated from pure DMU, PVA and DMU/HBSO as binders.

S/no	Parameter	DMU	DMU/HBSO	PVA	Standard(SON)
1	Viscosity(poise)	42.64	41.12	7.5	6 – 15
2	pH	8.62	7.89	8.80	7 – 8.5
3	Opacity	G	G	V.G	P
4	Drying time(min)	TD 15	18	17	20
		HD 87	86	75	120
5	Flexibility	P	P	P	P
6	Adhesion	V.G	G	G	P
7	Tackiness	P	P	P	P
8	Resistance to blistering	P	P	P	P
9	Stability	S	S	S	P
10	Chemical resistance	P	P	P	P
11	Gloss@ 85^{O}	20	18	18	16 – 50

P: Pass, TD: Touch dry, S: Stable, HD: Hard dry, V.G: Very good, G: Good

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In summary, even though DMU/HVO's have shown a lot of potentials as a binder in the formulation of EP, the viscosity is seen to be on the high side for this particular ratio that was used DMU/HCNO (70/30), DMU/HSO (60/40), DMU/HMSO (60/40), DMU/HBSO (60/40). This is far above the standard range. Therefore, it is advised that more of the HVO's should be used in order to obtain a viscosity result near that of the standard. This work may introduce a new EP binder into the coating industry.

Table 6: Comparison of EP formulated from different binders and with standard.

S/no	Parameter	DMU	DMU/HCNO	DMU/HSO	DMU/HMSO	DMU/HBSO	PVA	Standard
1	Viscosity (poise)	42.64	45.22	45.22	45.22	41.12	7.5	6 – 15
2	pH	8.62	8.77	8.77	8.77	7.89	8.80	7 – 8.5
3	Opacity	G	G	G	G	G	V.G	P
4	Drying time (mins)	TD 15	16	19	17	18	17	20
		HD 87	89	89	85	86	75	120
5	Flexibility	P	P	P	P	P	P	P
6	Adhesion	V.G	G	G	G	G	G	P
7	Tackiness	P	P	P	P	P	P	P
8	Resistance to blistering	P	P	P	P	P	P	P
9	Stability	S	S	S	S	S	S	P
10	Chemical resistance	P	P	P	P	P	P	P
11	Gloss@ 85^{O}	20	22	20	19	18	18	16 - 50

P: Pass, TD: Touch dry, S: Stable, HD: Hard dry, V.G: Very good, G: Good

The extractions of CNO, SO, MSO as well as BSO were carried out successfully. The various extracted oils were successfully epoxidized and hydroxylated. DMU was also successfully prepared. Copolymerization of DMU with the various hydroxylated vegetable oils was also successful. Various experiments were carried out on the pure VO's, EVO's, HVO's, as well as the copolymer resins. Parameters such as FTIR spectral analysis, viscosity, density, gel-time, turbidity, refractive index, formaldehyde emission, melting point, moisture uptake, elongation at break as well as solubility were carried out on the copolymers. The FTIR spectral analysis showed the presence of the basic functional groups present in the various copolymers. The viscosities were seen to increase with increase in the concentration of the HVO's in the copolymer because of the increase in molecular weight, while density was seen to decrease because vegetables oils have a lower density than the pure DMU.

Gel-time of the copolymers was observed to increase with increase in the concentration of HVOs, because it is responsible for the transition from liquid to solid state of the curing process. Turbidity as well as refractive index was seen to increase with increase in the concentration of HVOs. This is related to the gloss property of paint, as oils generally have a high refractive index. Formaldehyde emission was seen to decrease with increase in concentration of the HVO's due to the gradual reduction in the concentration of UF concentration in the copolymer. Moisture uptake was seen to decrease with increase in concentration of HVO's because of the hydrophobic nature of VOs. Elongation at break increased due to the increase in flexibility of the copolymer. Solubility was also observed to decrease with increase in concentration of the HVO's due to the hydrophobic nature of VOs.

Some physical properties of the EP formulated using the copolymer as binder were also carried out successfully. The various results obtained showed that DMU/HVO's show a great potential as a paint binder. Physical parameters such as viscosity and pH were seen to be above the standard values for most of the formulations, while opacity, drying time, flexibility, adhesion, tackiness, resistance to blistering, stability, and chemical resistance all passed the test. Gloss was within the standard range but higher than that of the pure EP. It was also observed that most of the tests carried out met that of the standard range. Therefore, it can be

concluded that DMU/HVO can be used as a binder to produce EP. This research work will greatly contribute to the development of the coating industry.

CONCLUSION AND RECOMMENDATION

Conclusion

This study evaluated DMU/HVO composites for water-resistant emulsion paint. Objectives achieved:

1. Extraction of CNO, BSO, MSO and SO.

2. Synthesis of DMU.

3. Epoxidation and hydroxylation of oils.

4. Copolymerization of HVOs with DMU.

5. Characterization by FTIR and physical tests.

6. Formulation and evaluation of emulsion paint.

Results showed: viscosity, gel-time, turbidity, refractive index and elongation increased to an optimum then decreased; density, melting point, moisture uptake and formaldehyde emission decreased with HVO loading. Solubility reduced at >40cm³. Paints met SON standards with better gloss and water resistance. Therefore, DMU/HVO copolymers are viable binders for water-resistant emulsion paint. Hydroxylated vegetable oils improved flexibility, water resistance and reduced formaldehyde emission of DMU without compromising coating properties. The binder combines performance of solvent-based paints with environmental safety of water-based systems.

RECOMMENDATION

For the development of a new paint binder from DMU/HVOs, the following are recommended:

i. The effect of viscosity on DMU/HVOs is an area of future research.
ii. UF with a lower mole ratio should be researched on, to further reduce UF ratio.
iii. Research on the use of other vegetable oils on DMU should be studied.

Contribution to knowledge.

This research work has the following contribution to knowledge:

1. New paint binders with very high potential for the coating industry have been introduced.
2. This research has added value to DMU and some vegetable oils.
3. This research largely confronts the problems associated with emulsion paint such as poor water resistance, gloss and durability of paint film. Thereby challenging the superiority of oil paint to emulsion paint.
4. If properly harnessed, the result from this study will highly minimize if not totally eradicate the problem of volatile organic compounds (VOCs) associated with oil paint and hence a reduction in the present-day global warming and climatic change.
5. This research can save foreign exchange for our country due to the potential reduction in the importation of paint binders for our coating industries.
6. It is a potential source of employment for our youths.

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