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Synthesis and Characterization of New Demulsifier from Natural Polymer

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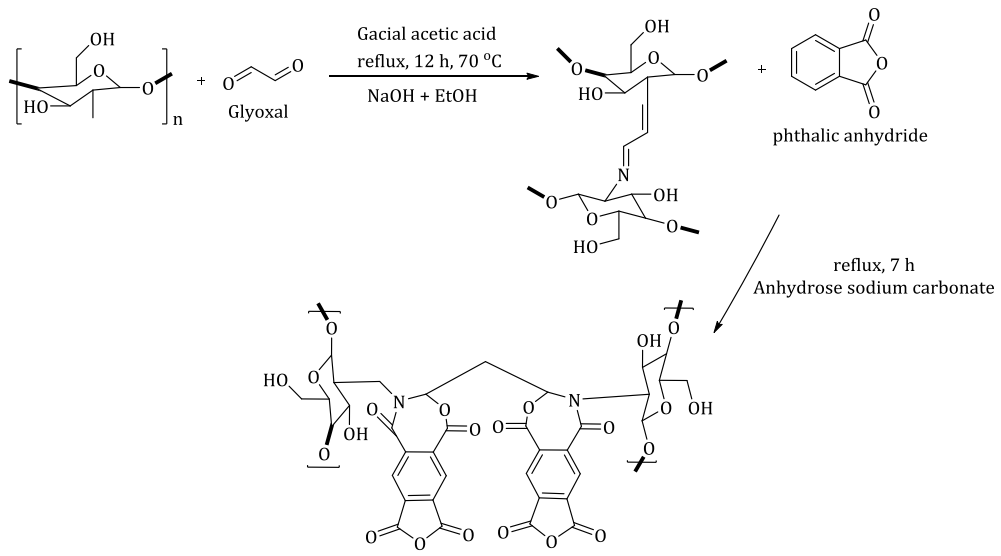
ABSTRACT

In this study, demulsifiers were prepared from Basra crude oil and water emulsions. This research included the preparation of new emulsifiers from natural polymers by forming a new flange base of chitosan with glyoxal. The prepared compounds were investigated using infrared spectroscopy and nuclear magnetic resonance spectroscopy as well as studied in terms of thermal stability using differential calorimetry technique. The prepared emulsifier was compared with a commercial emulsifier EC2134A, and it was shown that the prepared emulsifier is more efficient than the commercial emulsifier.

GRAPHICAL ABSTRACT



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Introduction

Demulsifers are substances used to break emulsification and carry different trade names and they are mostly polymeric materials with high molecular weights that work to equalize the effect of emulsifying agents and reduce the surface tension of water droplets. Further, they are surface active substances; however, the excessive usage of this substance can form emulsions that are more stable. Moreover, the separation of water from oil can be done using this substance because it is able to move through the oil phase to the boundary of the drop and in most cases, the demulsifier will neutralize the emulsifying agents and will help break the droplet membrane, helping the assembly. When the emulsifying agent is in an unstable state, breaking the droplet membrane causes the droplet size of water to be increased rabidly.

Many studies have been carried out on the demulsification and stability of the emulsions of crude oil, investigating more specifically the interfacial viscosity and tension effects and neglect of the lifetime, the interfacial elasticity, and the thinning rate of the oil film. Nevertheless, there is no certain demulsifer which has the ability to break down all types of emulsions of crude oil due to the complication of demulsification mechanism. In the current study, the demulsification of W/O synthetic emulsion has been investigated in several aspects such as life time and thinning rate of oil film as well as the oil-water interfacial properties with various demulsifers. Nowadays, there is a great demand on the usage of polymer-based and surfactants in order to enhance the recovery of oil, leading to form emulsions such as water in oil (W/O) or oil in water (O/W) [1, 2]. In fact, 80% of world production of crude oil is presented in forms of emulsified [3] and the greatest part produced crude oil is in form of W/O emulsion. When it comes to the factors that affect the composition of emulsion, then origin of the emulsion, the W/O ratio and natural emulsifier systems are the main factors [4]. Further, by comparing the heavy crude oil reserves with light and medium reserves, the former is considered to be more complicated, as many undesired merits are possessed by heavy crude oil such as high acidity and viscosity, also they tend to form stable emulsions. Thus, it is considered to be a main challenge for the industry of oil [5, 6]. The W/O emulsions with high stability can be formed because of the presence of asphaltenes, naphthenic acids, resins and fine solids as emulsifying agents for heavy crude oil reserves (Figure 1) [7, 8].

However, there are several economic problems that can be caused by water and crude oil emulsions including increasing the viscosity of oil, the growth of microorganism and the corrosion of pipelines. Therefore, it is an essential step to separate water from petroleum oil even before the refinement processes. It is worth mentioning that the prepared emulsion has the ability to be broken down chemically, electrically and mechanically [9]. Practically improving the combination techniques will end up increasing the efficiency of emulsification, whereas the most common used technique includes combining the chemical and heating demulsifiers [10].

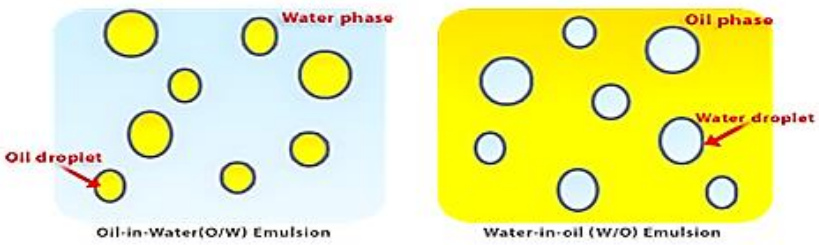


Figure 1: Emulsion Types

The chemistry of demulsifiers is based on several aspects such as phenolformaldehyde resins that are acid or base catalyzed, the epoxy resins, the polyamines, the poly ethyleneimines and the di-epoxides.

Emulsification mechanisms

The theory of surface tension involves occurring of emulsification when interfacial tension divided into two phases, whereas the theory of repulsion involves creating films by the emulsifying agent that cover one of the phases to form droplets that repel each other. Usually the emulsion forms when two unmixable liquids mixed together such as water and oil with aid of an emulsifier like phospholipid, protein and nanoparticle. However, types of formed emulsion can be determined according to the type of emulsifier that is used [11, 12].

Breaking down emulsions of oil nonpolar solutes or water polar solute consists of employing demulsifying agents. These agents are used in transformer fluids, rust and oxidation fluids and metal removal fluids as functional fluids. Berger and Hsu (2000) have explored destabilizing the (w/o) emulsion in the crude oil by using water soluble demulsifier rather than oil soluble demulsifier; they found that the emulsion could be destabilized using water soluble surfactant. On the other hand, polymers with different structures, molecular weight and HLB can be used for destabilizing the w/o emulsion through adding to the water phase. Molecules with low molecular weight and high hydrophilic moieties can demonstrate good abilities as demulsifying agent, which is often used to desalt and dehydrate crude oil.

Various concentrations of suspended matter and salts are present in crude, depending on where the oil located in. The majority of oilfields around the world have two different unmixable phases of water and crude oil, whereas stable emulsions of water-oil (W/O) can occur because of the interfacial active materials as well as the solid particles. Emulsion of crude oil causes operational problems as well as wastewater that is oily. Thus, demulsifying the formed emulsions is essential. There are different techniques to demulsification that are physical, biological and chemical. In chemical technique, the rate of film thinning is enhanced as well as lowering the stability of emulsion through using demulsifers classified into three categories according to their application and chemical structure [13, 14]: Ionic liquids, nanoparticles, polymeric surfactant. However, dendrimers are widely considered due to their effectiveness in different emulsions [15] Moreover, the modified polymeric surfactants as nanoparticles are more powerful than their counterpart that are not nanoparticles, as they show higher ability of breaking the emulsion but they are not researched enough. Various ethoxylated sugar-based amine surfactants have been synthesized and various factors that affect the performance of demulsification have been studied. Further, there are several factors that affect the efficiency of demulsification such as demulsifier concentration and temperature. Determination of the optimal dosage of demulsifers to break the emulsion depends on hydrophilicity and lipophilicity factors [16].

The stable emulsion such as water in oil (W/O) in petroleum industry is undesirable because it induces the steel corrosion, leading to the failure of pump, and considered as undesirable in refining operation. These emulsions are very stable due to the formation of stable films at water or oil droplets, producing either from crude oil components such as asphaltene, resins or from oilfield chemicals used for the different crude oil production operations. There are different demulsification techniques used to separate water from crude oil emulsions such as mechanical, thermal, electrical and chemicals demulsification methods. Because of its higher and rapid efficiency for demulsifying crude oil emulsion, chemical demulsification attract researchers' attention more than the other techniques [17, 18]. Further, different types of blends of nonionic surfactant can be used to formulate the chemical utilized in the demulsification processes. The chemical structure of these demulsifiers consists of two parts: The hydrophobic parts such as amide, alkyl fatty ester, alkyl phenol formaldehyde ether and polyoxypropylene, while the second part is hydrophilic such as polyoxyethylene. The surfactant should be able to adsorb water or oil droplet in order to take the place of the films of asphaltene and occupy the interface of water/oil. The used chemicals have some disadvantages such as being pollutant and expensive, thus, designing

new demulsifiers that are eco-friendly and economical is required [19, 20]. In fact, natural and artificial formation of oil and water emulsions can occur through washing by water in desalted unit [21, 22].

The range of content of the emulsion is between 0 to 5% by mass [23, 24]. In order to break the emulsion, an aqueous phase should be formed that contains the suspended matter as well as the salts, while the oil phase that represents the organic phase contains hydrocarbons. In fact, the water and salts can be problematic when comes the distillation columns unless it is removed [18]; additionally, it induces the corrosion in the transportation production as well as the crude oil refining and increases the pressure of the crude oil specifically when the water evaporates under exposure of high temperature and might eventually lead to explosion [25, 26]; thus, removing water thoroughly from the crude oil before the refining processes is an essential step that can be carried out using demulsifiers. Throughout this study, two demulsifiers have been investigated with high separation ability of the mixture of water and crude oil [27].

Schiff base

The azomethine group (>C=N-) is present in Schiff bases. Hugo Schiff originally described them in 1864 as condensation products of carbonyl compounds (aldehyd or ketone) with primary amines. The formation of Schiff base is caused by the reaction of primary amines with carbonyl compounds. All of these compounds have the azomethine group that possess a general formula R-HC=N-R_1, where R and R_1 can be aryl, alkyl, heterocyclic or cycloalkyl groups. From a structural standpoint, the formation process of Schiff' base (also called azomethine or imine) includes imine or azomethine group replacing the carbonyl group (>C = O) with a nitrogen that is an aldehyde or ketone analogue. Schiff bases also have antiviral, anti-inflammatory, antimalarial, antibacterial, antifungal, antipyretic and antiproliferative effects, among other biological actions. Various natural, naturally generated and unnatural substances including azomethine or imine groups. The biological activities of these substance can be attributed to the presence of these groups [28, 29].

Chitosan is one of the important renewable materials produced from *N*-acetylation of chitin that exist widely in the walls of fungi and plant cell as well as in the shell of fish. In addition, Chitosan is known for being highly biocompatible, biodegradable, and antifungal active material [30], attributed to the peculiar chemo-physical properties as well as biological activity, thus, there are many applications in that regard such as catalysis, pharmaceutical, food processing. Chitosan consists of mainly glucosamine (GlcN) and *N*-acetylglucosamine (Glc-NAc) in less quantity. It is worth mentioning that adequate efforts have been made to investigate the effect of hydroxyl and amino groups present in chitosan properties. On the other hand, imine groups can be formed when the active carbonyl groups react with amino groups in order to create Schiff base. In fact, physiological activity as well as antiphlogistic, antibactierial, and antiviral activities have been reported for these Schiff bases. However, the complexes of Schiff base can behave as catalysts, engendering a lot of research having been carried out on the complexation of Schiff base derived from chitosan such as the study focusing on the biological activity of chitosan Schiff bases and their complexes as well as carboxymethyl chitosan related to active hydroxyl and amino groups concentration in the chain of the molecule [31]. The influence of Chitosan Schiff bases on the kinetic of thermal decomposition was studied [32] by following procedure that is non-isothermal; as a result, the growth shows non-linear relationship affected by the extent of Schiff base substitution in the chitosan with respect to pre-exponential factor and activation energy [33, 34]. In this work, new demulsifies of Schiff base from chitosan with glyoxal have been prepared.

Materials and Methods

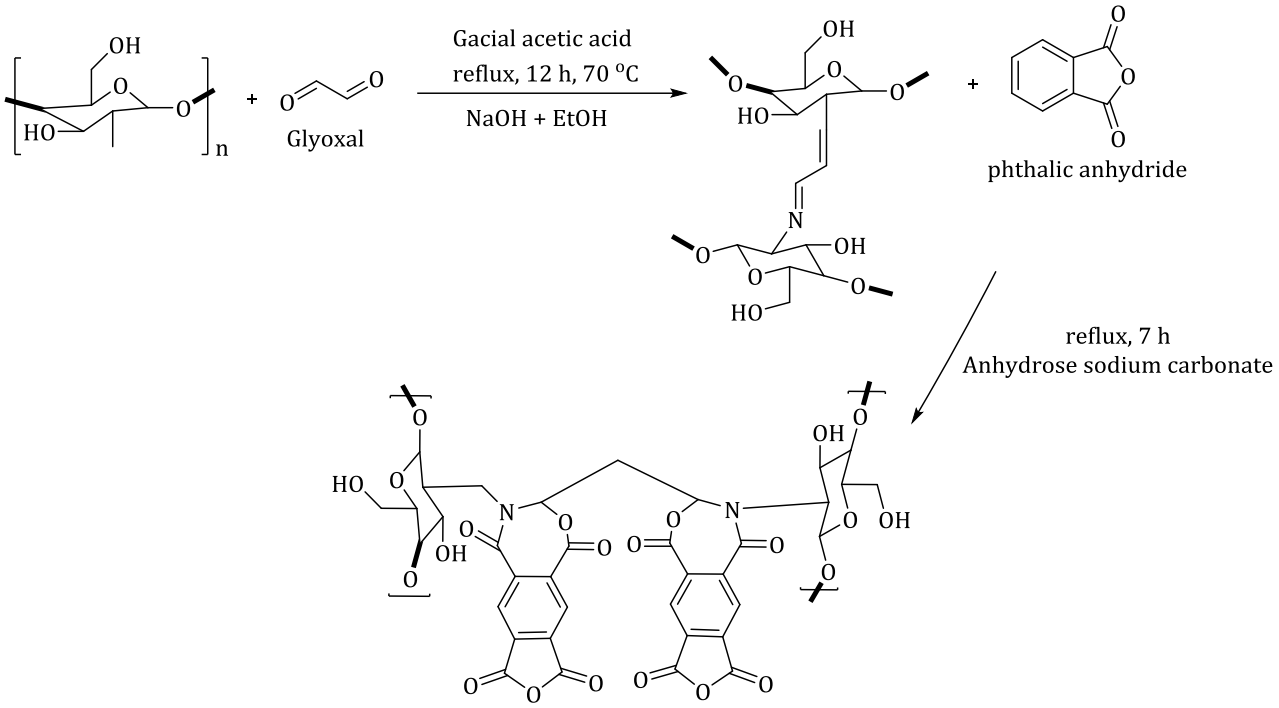
The materials applied included Chitosan (96%), glyoxal (98%) sodium hydroxide (99%), glacial acetic acid (99%), diethyl ether (98%), dimethyl sulfoxide (DMSO) (99%), and absolute ethanol (99%).

Preparation of Schiff base from (chitosan and glyoxal) {buta-1,3-diene ammonia ethyl ethyl [4,5-dihydroxy-6-(hydroxymethyl)-3-methyloxan-2-yl]methyl [3-amino-4,5-dihydroxy-6-(hydroxymethyl)oxan-2-yl]methylidene} (Chit-Gly-Schiff base)

A chitosan (1 g, 0.008 mol) in 50 mL of 1% glacial acetic acid was stirred at 30 °C for 2 h. A glyoxal (0.009 mol, 0.52 g) mixed with chitosan. The mixture was stirred and heated in a water bath at 70 °C for 12 h. 5% of sodium hydroxide drops was added until the precipitation of the desired compound. The transparent white precipitate was collected and washed with 10 mL diethyl ether to remove any remaining materials. The brown residue of polymer was obtained with a yield of 75%; the melting point was measured between 80-110 °C.

Synthesis of 1,3-oxazepine derivatives

A mixture of Schiff base (3.64 g, 0.01 mol) and 0.02 mol of phthalicanhydride were refluxed for 7 h in dry benzene. The solid product was filtered and recrystallized from ethanol. The products were washed with 10 mL diethyl ether and to remove any remaining materials, then they were subjected to filtering and drying at 60 °C using vacuum oven overnight. A solid, coffee-colored precipitate was obtained with a yield of 50% and the melting point was measured by 90-120.



Scheme 1: Mechanism reaction of Synthesis of 1,3-oxazepine derivatives

Preparing wet oil

Wet oil was prepared by mixing 5 mL of distilled water, with 1 g of sodium chloride (NaCl) and (70 ml) Basra crude oil using blending this mixture stirring for about two hours [35, 36].

The method of breaking emulsification

The process of breaking the emulsification was carried out according to the volumetric cylinder test, where 70 mL of oil emulsion (wet oil) was taken into a volumetric cylinder and different volumes of emulsifier were added to it from a concentration of 10-80 ppm dissolved in dimethyl sulfoxide. Then, the cylinder was closed and agitated by hand for a period of 10 min. and left; we intended to observe the amount of water separated with time at room temperature. After stabilizing the amount of separated water at a certain limit, the container cylinders of different sizes were placed in a bath containing hot water

(70 °C) and left to be pursued for the separated water quantity [37, 38].

Evaluation of the prepared demulsifier

0.1 g of Chit-Gly-Schiff base demulsifier was dissolved in 100 mL of dimethyl sulfoxide and mixed well to obtain a laboratory demulsifier. Different concentrations of demulsifier were added to the emulsion under study and at the room temperature, as shown in Table 1 representing the water amount that is separated from the demulsifier [39].

Table 1: Water amount that is separated from the demulsifier

Demulsifier (M1)	10 ppm	20 ppm	30 ppm	40 ppm	50 ppm	60 ppm	70 ppm	80 ppm
Temperature of room 20 °C	24 mL	25 mL	26 mL	27 mL	28 mL	29 mL	30 mL	31 mL
Hot water bath 80 °C	36 mL	38 mL	40 mL	41 mL	43 mL	45 mL	47 mL	48 mL
Commercial demulsifier 80 °C	20 mL	22 mL	24 mL	25 mL	26 mL	27 mL	28 mL	29 mL

Results and discussion

Oil emulsion was prepared using Basra crude oil with water, followed by adding salts to it to form water similar to the properties of oil wells water. As the mechanical drive, it works to dissipate the water in the oil as a result of high speed and after droplets are formed and fused; they are stable droplets [40].

Because alcohol can dissolve with membrane-forming compounds, the thin surroundings of the water droplets; this leads to the weakness of this membrane, hence reducing the stability of the emulsion. Alcohol acts also on the tearing of the micelles, which are aggregates of substances surface effective sphere, by reducing the charge density surface, so that the surface-active materials are in the form of free molecules that lead to an increase in its effectiveness towards breaking down emulsification [41].

Separation efficiency

Figures 2 and 3 show the effect of EC2134A and Cg on the efficiency of water separation with respect to time where the separation of water increases for Re-SOLC ®EC2134A to reach 60 min, then levels off with low rate of separation. However, after 100 min, the separation of 87 EC2134A was recorded as the best separation. Compound 2 exhibits an increase in the separation of water over time, as the rate of separation gets higher at the first 30 minutes and after that decreases dramatically. 85% was recorded as best separation for Cg for 100. Nevertheless, the threshold was 60 mins where both demulsifiers could not function for improving the separation beyond this time. The value of threshold was correlated with the concentration of demulsifier. Further, because of fully engaging of active chemical groups in the polymeric chain such as OH, C=O and COOH, slight increases in separation for Cg and EC2134A were observed after the 60 min., as these groups were not available for improving the separation.

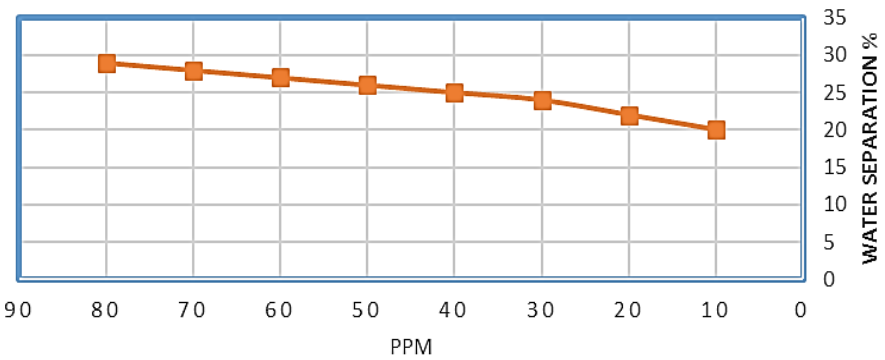


Figure 2: Different concentration of (Cg) influencing the water separation, where 15 vol% is representing the water phase and at 20 ⁰C

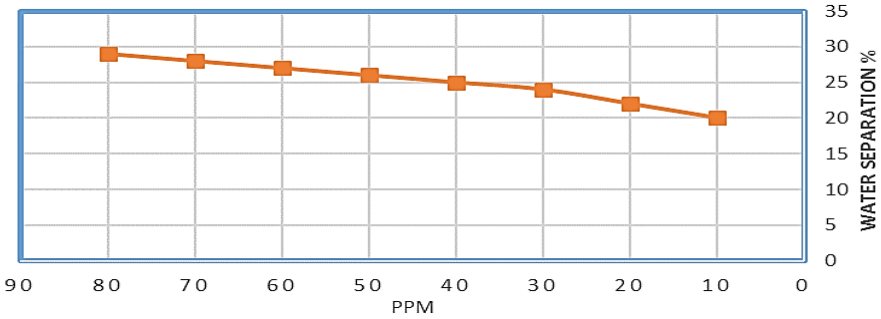


Figure 3: Different concentration of (Cg) influencing the water separation, where 15 vol% is representing the water phase and at 80 °C

The newly O-chitosan derivatives were prepared through the reaction of glyoxal with chitosan in distilled H_2O in acidic media. The FT-IR of derivative accounted for the presence of a big band at 3390 cm^{-1} referred to the stretch vibration of O-H, while 3209 N-H from the intra-and extra-molecular hydrogen bonding of chitosan molecules and a new band at 1705 cm^{-1} corresponded to C=O of ester (Figure 4).

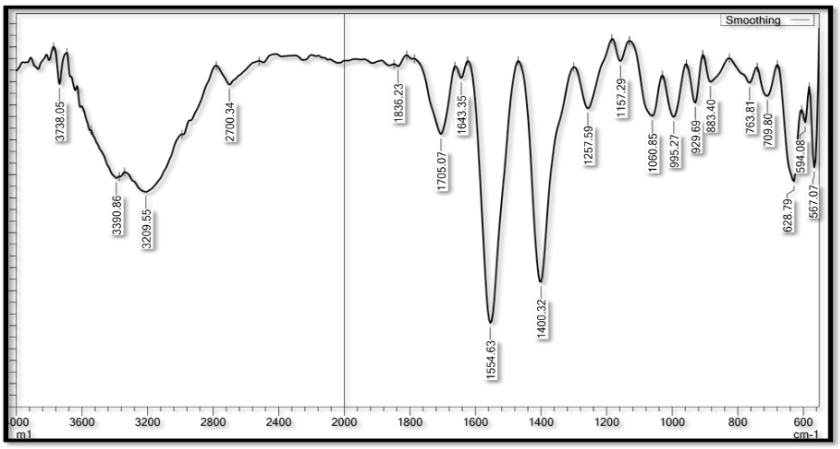


Figure 4: FT-IR of Chit-Gly-Schiff base

The synthesis of new derivatives starting from bis Schiff bases is evident in Scheme 1. New 1,3-oxazepine derivatives were prepared from substituted imine with phthalic anhydride.

Figure 5 on Cg shows bands at 1635 and 1705 cm^{-1} belonging to the C=O groups in the oxazepine ring 17 and bands around 1259 and 1143 cm^{-1}. The infrared spectra of the Chit-Gly-Schiff base show a medium intensity bands at the range of 3738 cm^{-1} related to hydroxyl groups stretching vibrations, a medium intensity band at 3209-3390 cm^{-1} that belongs to the NH_2 group, while the spectra of bands at 2700.34 cm^{-1} due to the stretching vibrations of -C-H saturated bonds, 1705 cm^{-1} medium the stretching vibrations of C=O and C=N for these compound appeared as a strong bands at 1554 cm^{-1}, while the C-N stretching vibrations appeared at 1257 cm^{-1}; likewise, the C-O stretching vibrations appeared at 1060 cm^{-1} of compounds spectrum. Finally, the bands at ~ 763 and 567 cm^{-1} were due to the bending vibrations of C-H aromatic and C-H alpiphatic, respectively (Figure 4). The infrared spectra of the compound 2 (Figure 5) show a medium intensity bands at the range of 3421 cm^{-1} pertaining to the stretching vibrations of hydroxyl groups, the low intensity of these bands due to the restriction by the large structure of the compound a medium intensity band at 3275-3155 cm^{-1} due to the NH_2 group, showing two bands of cm^{-1} that correspond to C-H aromatic as well as =C-H, respectively, while the spectra of Cg-2 show bands at 2927-2850 cm^{-1} that correspond to the stretching vibrations of CH_2-CH_3, 1705.07 cm^{-1}

medium band of stretch vibration of carbonyl group, whereas C=N stretching vibration of these compounds appeared as a strong bands at 1562 cm^{-1}. However, the stretching vibration of C-N emerged at 1238 cm^{-1}, as well as the stretch vibration of C-O appearing at 1010 cm^{-1} of compounds spectrum. Finally, the bands at ~ 794 and 640 cm^{-1} correspond to the bending vibrations of C-H aromatic and C-H aliphatic, respectively.

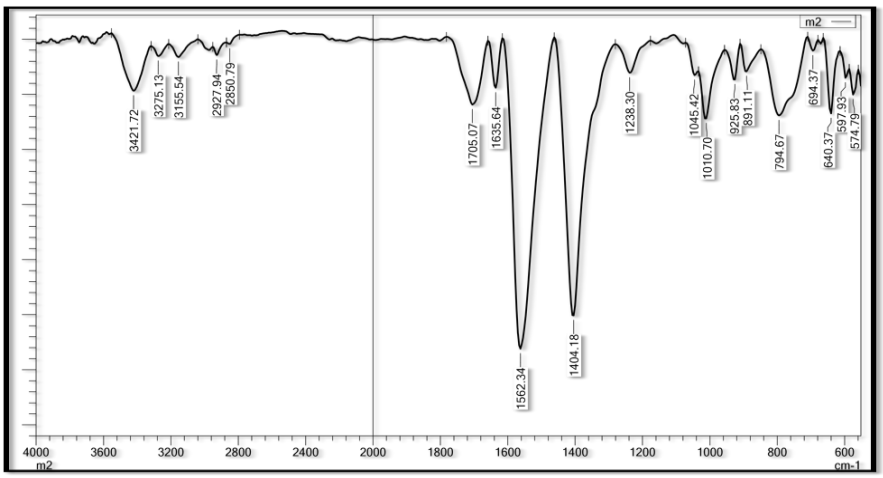


Figure 5: FT-IR of Cg-2

^{1}H-NMR of Chit-Gly-Schiff base

The ^{1}H-NMR of Chit-Gly-Schiff base shows a broad signal at 4.76 ppm, which indicates H1 proton (Figure 7). Furthermore, the spectrum showed multiplet signals at a range of 3.80-4.05 ppm corresponding to H3–H6 protons. The spectrum also showed a relatively broad singlet signal belonging to H2 protons at 3.54 ppm. Additionally, the spectrum shows the characteristic signals of the added molecule (benzaldehyde) as two multipletes at the range 7.80-7.89 ppm and 7.20-7.26 ppm attributed to the Ha and Hb/Hc protons of the aromatic ring, respectively. Moreover, the spectrum exhibited a singlet signal at 8.46 ppm attributed to the CH proton of azomethine group. The high intense signals at 2.50 and 3.37 ppm represent the protons of DMSO and H_2O, respectively. ^{1}H-NMR spectrum in DMSO of Schiff base [I] showed a sharp signal at 1.5 ppm for one proton attributed to the CH=N.

Thermo gravimetric analysis of this compound showed four stages of weight loss, which were as follows (Figure 8):

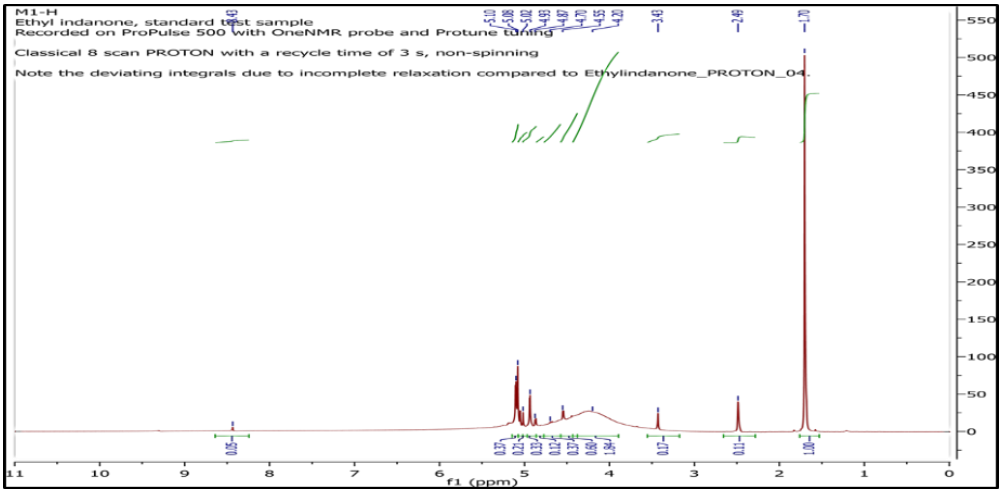


Figure 7: ^{1}H-NMR of Chit-Gly-Schiff base

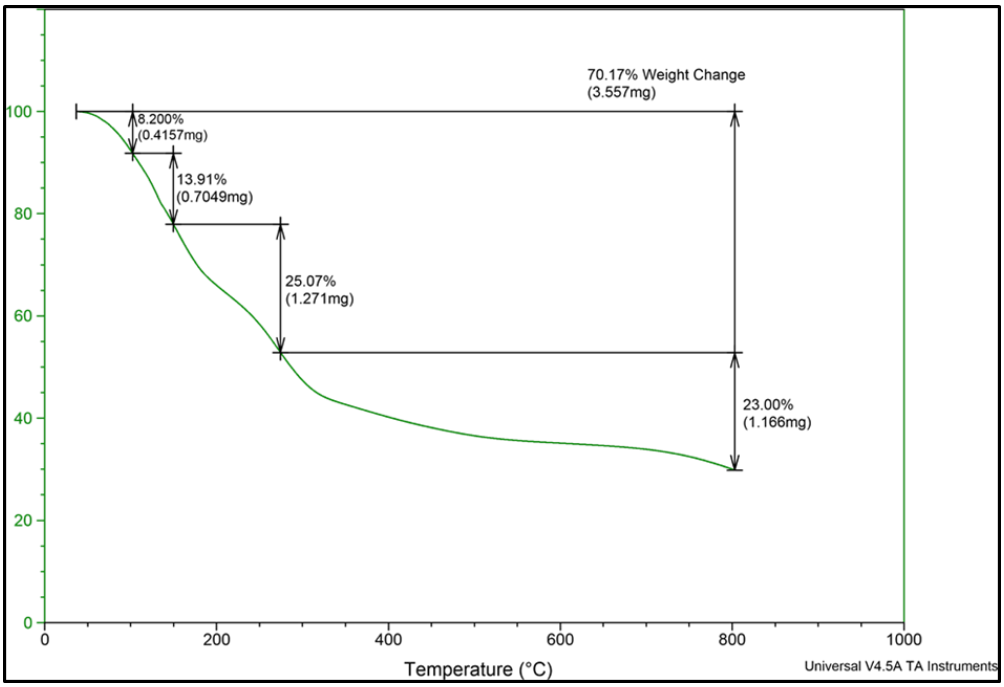


Figure 8: Thermal analysis of Chit-Gly-Schiff base

1) The first stage, which was at 140.23 °C, is attributed to the evaporation of water present in the form of moisture or crystallized in the compound, with a weight loss 12%. 2) The second stage, which was at 269.98°C, is attributed to the decomposition of chitosan bonds with glyoxal and decomposition of benzaldehyde, with a weight loss 17%. The wt.% of loss was relatively small; therefore, this stage can represent the proportion of the reacted benzaldehyde. 3) The third stage included a loss of 23.5% at 475.10 °C, which is attributed to the decomposition of the bonds forming chitosan. 4) The fourth stage, which is the last stage, included the largest weight loss 33.7% at 807.39 °C, which indicates the continued degradation of chitosan and the emission of carbon dioxide, while the remaining wt.% is carbon waste.

Conclusion

According to the current study aiming an applicable and empirical study, there are several conclusions that can be drawn; firstly, increasing the separation time of the tested demulsifers leads to increase the separation of water. Secondly, the concentration of demulsifer can play an important role in limiting the separation of water. Lastly, increasing the dosage of dimulsifers will increase the efficiency of separation of water accordingly.

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Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to responsible for all the aspects of this work.

Conflict of Interest

We have no conflicts of interest to disclose.

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