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Modification of a biopolymer, characterization and application

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Dedications

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Sarra

Dedications

With gratitude to Allah, who illuminated my path and granted me strength and patience, I dedicate this work — the fruit of years of effort and perseverance — to those whose presence was a steadfast support, a constant source of inspiration, and a guiding light through every step.

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Summary

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Symbols and Abbreviations List

A: Absorbance.

AM: Acrylamide.

APS: Ammonium persulfate.

BN: Bentonite.

BHT: Butylated hydroxytoluene.

C: Concentration.

CG: kappa carrageenan.

DPPH: 1,1-diphenyl-2-picrylhydrazyl.

DSC: Differential scanning calorimetry.

E%: Inhibitor efficiency.

EC_{50}: Effective concentration.

-g-: Graft.

HCl: Hydrochloric acid.

ι-carrageenan: Iota-carrageenan.

inh: Inhibitor.

IR: Infrared spectroscopy.

κ-carrageenan (κ-CG): Kappa-carrageenan.

λ-carrageenan: Lambda-carrageenan.

m_i: Initial mass.

m_f: Final mass.

Δm: Weight loss.

NaOH: Sodium hydroxide.

NTU: Nephelometric turbidity unit.

PAM: Polyacrylamide.

pH: Hydrogen potential.

S: Surface.

T: Temperature.

TGA: Thermogravimetric analysis.

θ: Surface coverage.

t: Time of immersion.

TU_0: Initial turbidity.

TU: Turbidity.

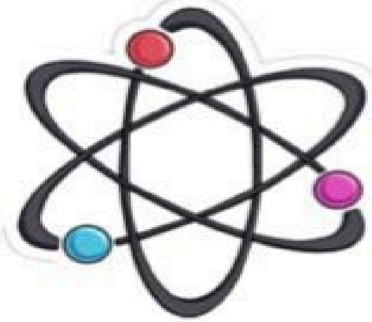
UV-Vis: Ultraviolet-Visible spectroscopy.

V: Volume.

W_{corr}: Corrosion speed without inhibitor.

$W_{(corr\ (inh))}$: Corrosion speed with inhibitor.

XRD: X-ray diffraction.



GENERAL INTRODUCTION

In various industrial sectors, a wide range of chemical substances is utilized to meet specific needs, including water treatment, corrosion prevention, and food preservation. While their effectiveness is often proven, their use raises increasing concerns regarding toxicity, sustainability, and environmental impact [1-3].

In the field of water treatment, chemical coagulants such as ferric chloride ($FeCl_3$) and alum—a double sulphate of potassium and aluminium with the formula $KAl(SO_4)_2 \cdot 12H_2O$—are widely employed due to their ability to effectively remove turbidity, heavy metals, and both organic and inorganic substances, even at low concentrations. However, despite their efficiency, these products have several major drawbacks: high cost, potential toxicity, and environmental impact. In particular, residual aluminium that may remain in treated water is suspected to be linked to serious neurological disorders, including Alzheimer's disease, dementia, encephalopathy, Down syndrome, and Parkinson's disease. Additionally, these coagulants can alter the pH of the treated water, be corrosive or carcinogenic, and produce large amounts of non-biodegradable sludge, the disposal and management of which remain costly and environmentally unfriendly [1].

Similar issues are encountered in the oil and gas industry, particularly in transport infrastructures such as subsea pipelines. Various chemical compounds are used to combat corrosion, yet each presents significant limitations. For instance, hydrate inhibitors like methanol are highly volatile, leading to considerable economic losses due to evaporation when they come into contact with gas. Diethylene glycol, though less volatile, creates challenges related to emulsification, solubility, and regeneration. As for surface-active agents (surfactants), used in enhanced oil recovery to improve water injection, they are often expensive and only partially effective, as their adsorption onto rock surfaces reduces their performance. Moreover, insulating coatings applied to pipelines (such as bitumen, epoxy, cement, fiberglass, and paints) may deteriorate under harsh conditions or even promote corrosion if poorly applied or damaged. [2]

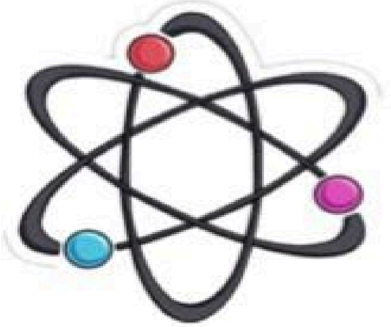
In the agri-food sector, synthetic antioxidants are commonly added to processed foods to slow lipid oxidation and extend shelf life. However, several studies have highlighted their potential toxic and carcinogenic effects, leading to stricter regulations. These compounds also suffer from various functional limitations, such as low solubility in water, reduced effectiveness at high temperatures or under UV radiation, and a tendency to accumulate in adipose tissue. For example, gallates like propyl gallate can react with iron ions to form dark precipitates and have been associated with liver toxicity. Similarly, BHT may generate free radicals under certain conditions, limiting its use. These drawbacks, combined with declining industrial interest, are prompting a growing shift towards natural alternatives that are considered safer and more compatible with public health. [3]

In response to these challenges (health-related, environmental, or economic), an increasing number of research efforts are now focusing on the use of natural, biodegradable, and renewable materials, particularly polymers derived from biomass. Conventional polymer production relies heavily on fossil resources and hazardous reagents, contributing to pollution and waste accumulation. To address this, the industry is embracing the principles of green chemistry, favoring renewable raw materials, safer transformation processes, and the design of degradable materials. [4]

Polymers—whether natural (such as rubber or DNA) or synthetic (like polyethylene or PVC)—are macromolecules composed of repeating monomer units, which endow them with diverse properties, including strength, flexibility, and insulation. These qualities make them essential across a wide range of applications in health, agriculture, electronics, and industry [5].

In this context, our study focuses on carrageenan, a polysaccharide extracted from red algae, known for its multiple biological activities, including antitumor, antiviral, antioxidant, and anti-inflammatory effects. Despite its potential, its limited water solubility restricts its range of applications. To enhance its physicochemical properties and improve its usability in aqueous formulations, we have undertaken a chemical modification via acrylamide grafting [6-8].

This memoir is divided into three main chapters. The first chapter provides an overview of the literature on k-carrageenan, acrylamide, grafting methods, and the various applications of polymers in the fields of water treatment, corrosion protection, and antioxidative properties. The second chapter provides a detailed description of the equipment used and the experimental methods employed, including the copolymer synthesis protocols and the physical-chemical characterization procedures implemented. Finally, the third chapter presents and analyses the obtained results, as well as interprets the data resulting from the characterization and testing of the synthesized copolymers.



CHAPTER 01:

BIBLIOGRAPHICAL RESEARCH

1. General introduction to carrageenan:

1.1. Definition:

A family of naturally occurring, water-soluble, sulphated galactans extracted from red seaweeds is known by the general term "carrageenan” [9].
The sulphated groups are chemically combined via ester bonds to the carbon atoms C-2, C-4, or C-6 of individual galactose residues. The quantity of $-O-SO_3-$ in sulphated polysaccharides can be significant and differs in the range of 0-41% (w/w), creating highly charged negative polymers. Carrageenans are straight-linear, partly sulphated galactans that are mostly made up of repeated dimers of α (1------4)-linked D-galactopyranose or 3,6-anhydro-D-galactopyranose residue and a β(1-------3)-linked D-galactose residue [9].
There are various carbohydrate residues (including xylose, glucose, and uronic acids) and substituted elements (such as methyl ethers) besides the primary sugar residues, D-galactose and 3,6-anhydro-D-galactose, and sulphate [9].

CGs have demonstrated several possible medicinal properties, including immunomodulatory, anticoagulant, anticancer, and antihyperlipidemic effects [10].

Each of the proportions of carrageenan is soluble in water but insoluble in fats, oils, or organic solvents. Their water solubility, however, mostly depends on the number of sulphate groups (which are extremely hydrophilic) and the cations that are linked to them. Although additional ions can also exist at lesser frequencies, sodium, potassium, calcium, and magnesium are the primary ionisable cations found in carrageenans [11]. The functional characteristics of carrageenans are influenced by their structure [9].

The food industry uses carrageenan as a thickening, gelling, emulsifying, and stabilising agent. Due to its numerous applications as a polysaccharide, it also finds uses in non-food industries, such as printing, textiles, cosmetics, and pharmaceuticals [12].

Figure 1: *Carrageenan polysaccharide texture (white powder).*

1.2. Sources and types of carrageenan:

Red algae, or Rhodophyta, are responsible for the production of carrageenan in seaweeds [9].

Figure 2: *Rhodophyta red seaweeds.*

Figures 3, 4, and 5 show the architecture of the three most common CGs: kappa (κ), iota (ι), and lambda (λ) [13]. Cottonii produces a consistent κ-carrageenan after alkali treatment. Eucheuma denticulatum (trade name Spinosum) produces ι-carrageenan when extracted with alkali. Sporophytic plants produce λ-carrageenan [9].

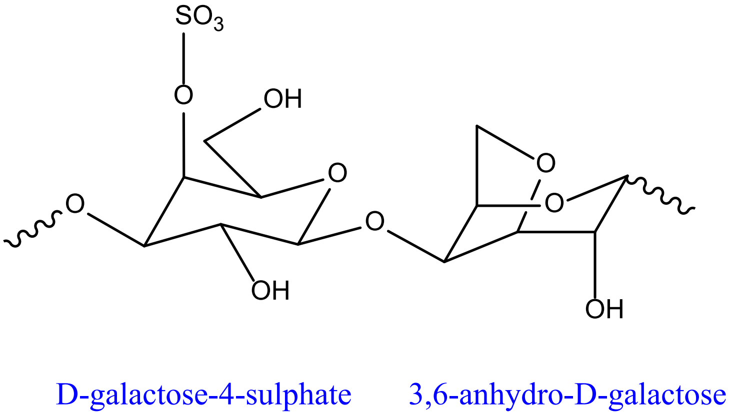
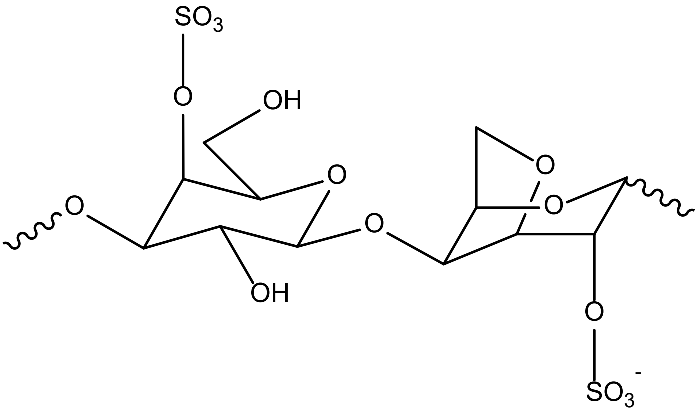
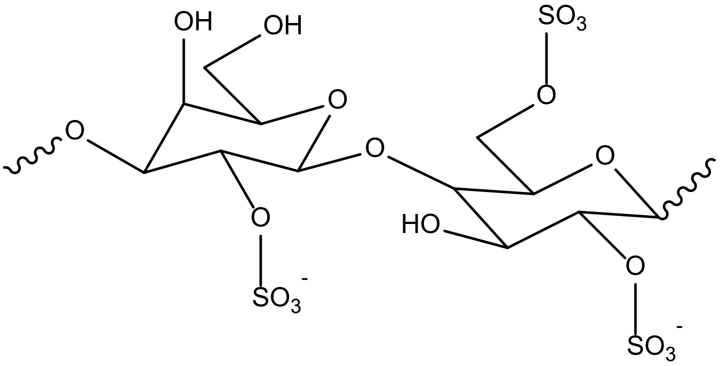


Figure 3: *Chemical structure of Kappa Carrageenan (κ).*



D-galactose-4-sulfate 3.6-anhydro-D-galactose-2-sulfate

Figure 4: *Chemical structure of Iota Carrageenan (ι).*



D-galactose-2-sulfate D-galactose-2.6-disulfate

Figure 5: *Chemical structure of Lambda Carrageenan (λ).*

In our study, we focus on kappa carrageenan for its unique properties.

1.3. Studies carried out on the modification of kappa carrageenan:

Synthesis, characterisation and applications of graft copolymer (k-carrageenan-g-vinylsulfonic acid):

This article describes the synthesis and characterisation of κ-carrageenan-g-vinylsulfonic acid. This graft copolymer was produced via polymerisation in a nitrogen atmosphere with a redox system (potassium peroxymonosulfate and malonic acid). The copolymer's characteristics, including its ability to swell in water, its ability to adsorb metal ions, and its flocculation effectiveness for treating wastewater from coal mines, were examined. Vinylsulfonic acid was successfully grafted onto κ-carrageenan, increasing its thermal stability and resistance to biodegradation. Additionally, the copolymer exhibits increased water retention capacity and better flocculation and metal ion adsorption efficiency, suggesting potential uses in a range of industrial domains.[14]

pH-responsive interpenetrating network hydrogel beads of poly(acrylamide)-g-carrageenan and sodium alginate for intestinal targeted drug delivery: Synthesis, in vitro and vivo evaluation:

In this work, pH-sensitive interpenetrating network hydrogel (IPN) beads made of sodium alginate (SA) and poly(acrylamide)-g-carrageenan (PAAm-g-CG) are synthesised and evaluated for the targeted release of ketoprofen in the intestine. Ionotropic gelation and covalent cross-linking were used to create the beads. The findings demonstrate that the beads' swelling and drug release behaviour are highly impacted by pH, with the most release occurring in alkaline media (pH 7.4) and the least in acidic media (pH 1.2). These beads enable controlled drug release in the intestine while reducing gastric adverse effects like ulcers and bleeding, according to in vitro and in vivo investigations. This work demonstrates that these hydrogel beads are excellent delivery vehicles for non-steroidal anti-inflammatory medications (NSAIDs) and peptides.[15]

Modified carrageenan.5. Preparation, swelling behaviour, salt- and pH-sensitivity of partially hydrolysed crosslinked carrageenan-graft-polymethacrylamide superabsorbent hydrogel:

A superabsorbent hydrogel based on κ-carrageenan grafted to polymethacrylamide (κC-g-PMAM) is synthesised and studied in this research. Alkaline hydrolysis is then performed to produce a partially hydrolysed hydrogel (H-κC-g-PMAM). Conditions for hydrolysis and grafting were adjusted to optimum swelling capacity, which may reach 435 g/g. Because of the masking effect of charges and ionic cross-linking, the hydrogel exhibits sensitivity to pH and salts and a decreased ability for swelling when multivalent cations are present. The hydrogel is a promising option for controlled drug release applications because it also shows reversible swelling/deswelling behaviour as a function of pH. Kinetic experiments reveal that it swells more quickly in water than in saline solutions.[16]

Water soluble graft copolymer κ-carrageenan-g-N-vinylformamide: Preparation, characterisation and application:

This article describes the synthesis and characterisation of κ-carrageenan-g-N-vinylformamide, a water-soluble graft copolymer that was produced via radical initiation using the malonic acid/potassium persulfate redox system. By adjusting reagent concentrations, grafting characteristics such as grafting ratio, add-on, and conversion were optimised. When compared to native κ-carrageenan, the graft copolymer exhibited improved swelling properties, metal ion sorption, flocculation, and resistance to biodegradation. This hybrid material may find use in controlled drug release and wastewater treatment.[17]

Free radical graft copolymerization of N-vinyl-2-pyrrolidone onto k-carrageenan in aqueous media and applications:

The creation and applications of a grafted copolymer that combines N-vinyl-2-pyrrolidone (NVP) and k-carrageenan. Using a redox pair of peroxymonosulphate (PMS) and malic acid in an aqueous environment, the copolymer graft is synthesised, with reaction conditions optimised to maximise the grafting rate. The improved properties of the copolymer such as its resistance to biodegradation, stronger ability to swell in water, increased sorption of metal ions, and improved flocculation properties.[18]

Grafting of acrylamide onto kappa-carrageenan via γ-irradiation: Optimisation and swelling behaviour:

Synthesis of a superabsorbent hydrogel using kappa-carrageenan (κC) and acrylamide using gamma radiation. The resulting hydrogels can absorb large amounts of water and have potential uses in a variety of fields, including personal hygiene, agriculture, and medication release systems.[19]

Simultaneously improved strength and toughness in κ-carrageenan/polyacrylamide double network hydrogel via synergistic interaction:

Producing hybrid double-network (DN) hydrogels by combining a covalently reticulated polyacrylamide (PAAm) network with a physically reticulated κ-carrageenan network. The use of a K^+/Ca^{2+} cation mixture simultaneously increases the hydrogel DN's strength and resistance due to a synergistic effect. Additionally, the hydrogel DN reticule K^+/Ca^{2+} has a high capacity for recovery at ambient temperature as well as the ability to thermally self-cicatrize. The suggested synergistic interaction strategy provides an alternative method of creating DN hybrid hydrogels with desired mechanical and functional properties.[20]

Lectin-functionalized carboxymethylated kappa-carrageenan microparticles for oral insulin delivery:

The use of lectin-functionalized kappa-carrageenan carboxymethylated microparticles for oral insulin delivery. The ability of these microparticles to enhance intestinal absorption and shield insulin from degradation in the gastrointestinal tract. To improve intestinal mucoadhesion, the microparticles were manufactured by ionisation and functionalized with lectin. The oral administration of insulin encapsulated in these microparticles caused a prolonged hypoglycemic effect in diabetic rats, suggesting their potential as an oral insulin administration system. The microparticles have demonstrated a good release profile and low toxicity.[21]

2. General introduction to acrylamide and polyacrylamide:

The industrially created α,β-unsaturated (conjugated) reactive substance Acrylamide (2-propenamide) is a crystalline solid with a melting point of 84.5 °C [22]. At 30°C, it is soluble in a variety of solvents, including water, methanol, ethanol, acetone, ethyl acetate, chloroform, benzene, and heptane [23]. Acrylamide is created when acrylonitrile is hydrated with sulfuric acid monohydrate at 90-100 °c [23]. While foods loaded with carbohydrates and cooked at temperatures above 120°C are exceptionally rich in acrylamide [24].

Polyacrylamide is a homopolymer of the same acrylic units [25]. In many different fields, including the paper and textile industries, soil conditioners, mineral processing, and cosmetics, acrylamide and polyacrylamide are frequently utilized [22], for regenerative medicine, PAM hydrogels are used as scaffolds for cell culture because they offer a soft matrix that replicates the characteristics of natural tissue [26]. Industrial wastewater can be effectively treated by copolymers of acrylamide, which also improve coagulation processes and lower turbidity [27].

Numerous test animals develop peripheral neuropathy as a result of repeated exposure to the highly neurotoxic acrylamide monomer [28]. The acrylamide monomer is easily breathed and quickly absorbed through healthy skin. It is widely accepted that the acrylamide monomer content of polyacrylamide is the primary toxicological concern [28]. According to reports, polyacrylamide loses all of its hazardous properties when used for grouting [28].

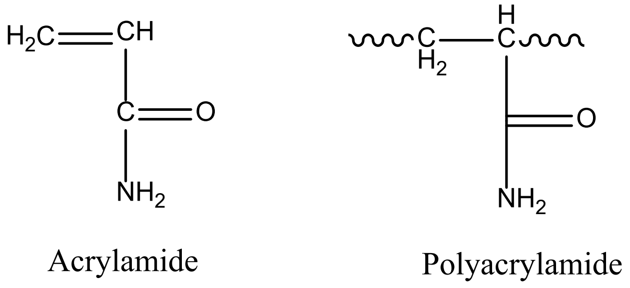


Figure 6: *Chemical structure of acrylamide and polyacrylamide.*

Figure 7: *Acrylamide texture (white powder).*

3. Clays:

3.1. Kaolin:

The natural modification of feldspar and other silicate minerals produces kaolinite, an abundant clay mineral resource with a layered silicate structure. This kind of aluminosilicate is made up of Si-O tetrahedral and Al-(O, OH) octahedral joined by a common oxygen atom [29]. Its chemical structure is $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ [29]. The benefits of kaolinite include high specific surface area, environmental friendliness, and exceptional stability [30], safe [31], low-cost clay mineral that is harmless [31], and an abundant silicate in soils and sediments [31].

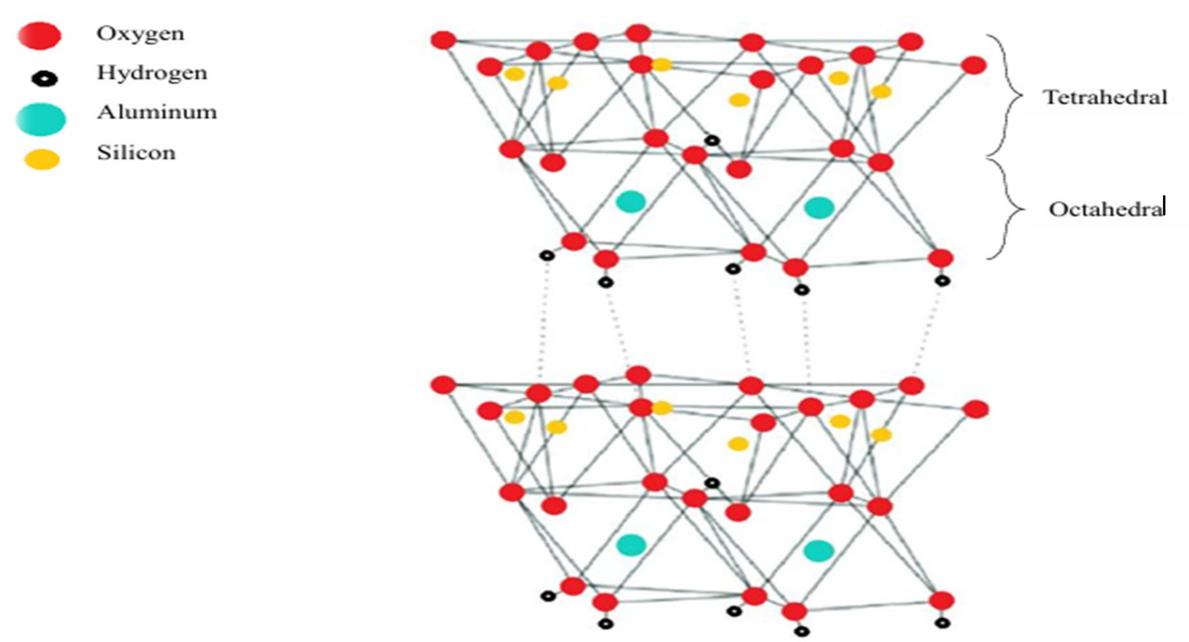


Figure 8: *Chemical structure of Kaolin.*

3.2. Bentonite:

Bentonite is primarily derived from volcanic cinders and is mostly composed of smectites, the majority of which are montmorillonite [32]. Montmorillonite is a hydrated aluminium silicate that contains trace amounts of alkaline earth and alkaline metals. On a structural level, it is made up of two basic components: an octahedral aluminium couch and a tetrahedral silica sofa. The two tetrahedra of silica that make up the cellular unit of montmorillonite include an octahedron of aluminium. Because this network is negatively charged, cations like calcium and sodium can adhere to its surface. The fact that these cations can easily change gives montmorillonite unique properties, such as the ability to absorb and retain ions [33].

Bentonite is classified into two types: "swelling" clays (heavy in sodium) and "non-swelling" clays (high in calcium) [34].

Because of its remarkable physical and chemical properties, bentonite has a wide range of industrial uses. It is especially used in animal litter, feed fluids, foundry sands, and iron mine pellets. Sodular bentonite is valued for its viscosity and ability to expand, whereas calcified bentonite is mostly used as an absorbent. She also works in the production of adhesives, aerosols, food additives, cosmetics, catalysts, and etching materials. It may be found in a wide range of industries, including agriculture, chemistry, paper, paints, plastics, pharmaceutical products, and even waste and water treatment. It is a polyvalent material with several industrial uses [35].

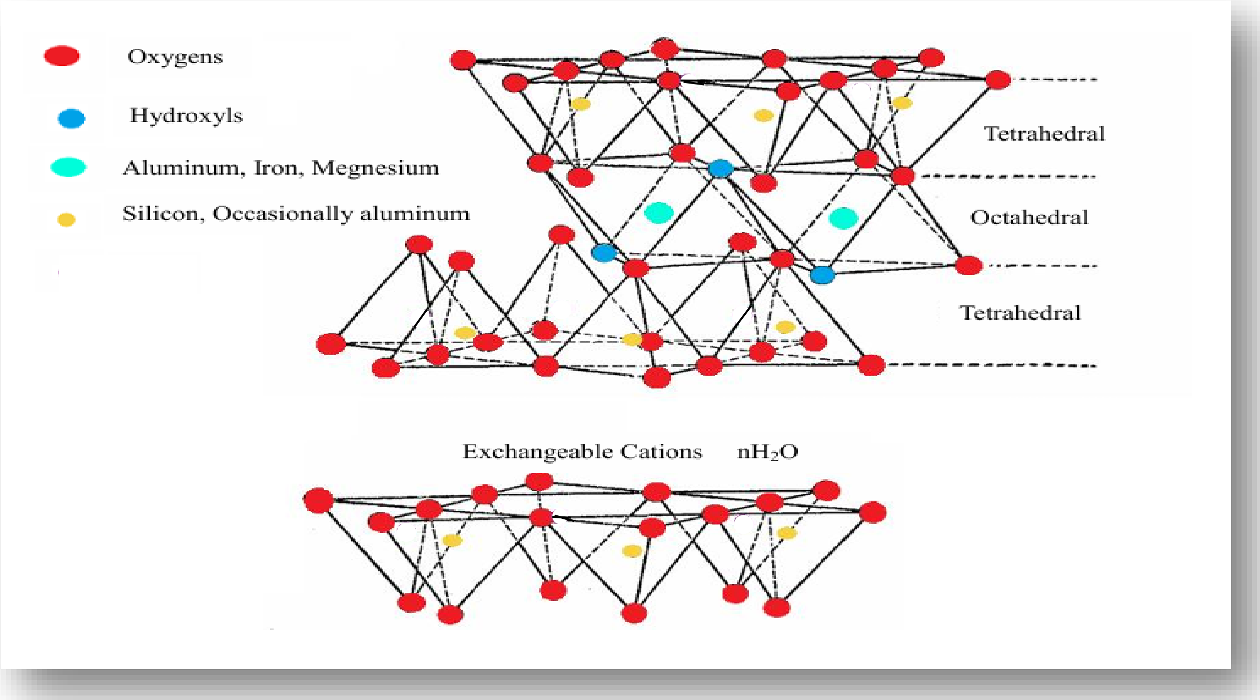


Figure 9: *Chemical structure of Bentonite.*

4. Generality regarding water treatment:

Water treatment is a crucial procedure aimed at purifying water for various uses, such as human consumption and environmental disposal [36]. The primary goal is to remove a wide range of contaminants, such as suspended materials and particles [37], [38], organic and inorganic dispersions [32], [33], micropollutants like pesticides and pharmaceutical products [36], pathogenic agents like bacteria, viruses, and protozoa [38], as well as substances that can produce tastes and odors [36].

There are several techniques for treating water. Their major techniques are:

4.1. Sedimentation:

Sedimentation is the process by which suspended particles are separated from one another by gravitational decantation. She plays an important part in water treatment. In water purification stations, sedimentation allows water turbidity to be reduced to very low levels, frequently in conjunction with coagulation to speed up the development of flocs that decompose quickly [39].

4.2. Filtration:

Oil, grease, and organic compounds are frequently removed from generated water using filtration technology. A variety of media, including sand, gravel, anthracite, walnut shell, and others, can be used to achieve filtration [37].

4.3. Adsorption:

Adsorption is a process where a material (the adsorbent) has a substance (the adsorbate) adhere to its surface, such as active carbon, moving from a liquid phase to a solid surface. This can be a physical process where the adsorbent is held in place by weak forces or a chemical process where a chemical bond forms between the adsorbent and the adsorbate. The nature of the material, environmental factors (such as temperature and pH), and the properties of the molecules being adsorbed all affect how effective the adsorption is [36].

4.4. Membranes:

Membranes, which are microporous sheets with certain pore ratings, are used to separate fluids from their constituent parts. Four recognised membrane separation techniques exist: reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF), and microfiltration (MF) [40].

4.5. Coagulation and flocculation:

The submicron particles that cause turbidity in water are colloidal and have a negative surface charge that prevents them from adhering and reorganising into larger, easier-to-get-rid-of particles. The use of chemical coagulants, such as metal salts (ferric or aluminium sulfate), is required to neutralise the charge of these stable particles. These coagulants work by a variety of mechanisms, such as double-layer compression, charge neutralisation, and flocculation by balayage. After being neutralised, the particles reorganise to form flocs, which makes their removal easier. The two main steps of flocculation are particle percutting and agglomerator formation. Perikinetic (by diffusion) and orthokinetic (by fluid movement) flocculation are two of the mechanisms that depend on this process. Factors including particle size, pH, and mixing speed affect the process's effectiveness, which also varies depending on the water supply [36].

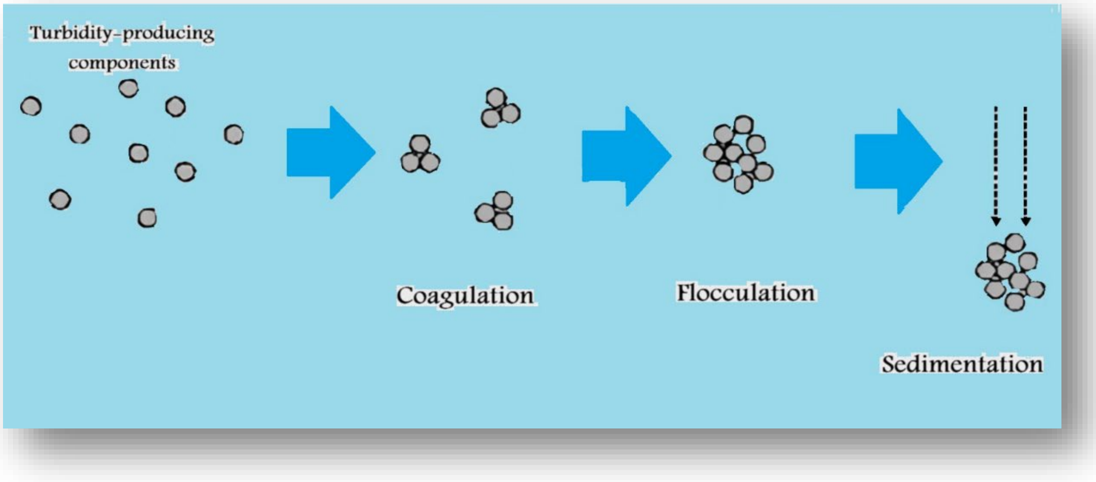


Figure 10: *Coagulation, Flocculation, and Sedimentation Processes.*

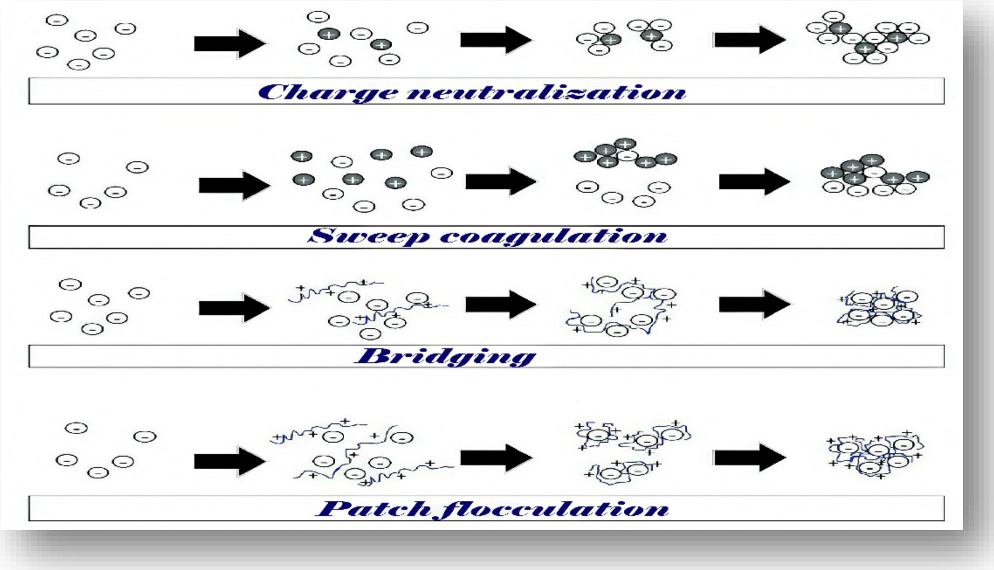


Figure 11: *Mechanisms of coagulation-flocculation.*

4.6. Disinfection:

Disinfection of water aims to eradicate pathogenic microorganisms to prevent water-related diseases, which are frequently caused by bacteria, viruses, and protozoa spread by the faecal-oral route. Introduced in England in 1904, this technique is essential to the safety of drinkable water. Chemical products like chlorine or ozone can be used to disinfect, or physical methods like heat, light, or microfiltration can be used and applied [36].

Our study focused on the coagulation-flocculation method.

5. Corrosion:

5.1. Definition:

An irreversible interfacial response between a material (such as metal, ceramic, or polymer) and its surroundings is called corrosion, and it can lead to the material's consumption or breakdown into an environmental component. Corrosion frequently, but not always, has negative impacts on how the material in question is used. The term "corrosion" does not refer only to physical or mechanical processes like melting or evaporation, abrasion, or mechanical fracture [41].

5.2. Types of corrosion:

5.2.1. Wet corrosion:

The term "wet corrosion" describes the electrochemical deterioration of metals that takes place when they come into contact with liquid electrolytes or dampness. Water is necessary for the formation of galvanic cells and the transport of ions, which facilitates this process. The pace of wet corrosion can be greatly influenced by variables like temperature, pH, and the presence of corrosive substances (such as salts or acids), which frequently results in rusting, pitting, and crevice corrosion [42], [43].

5.2.2. Dry corrosion:

Dry corrosion, also known as atmospheric corrosion, takes place in environments where moisture is absent, and metals are exposed to gases. This type of corrosion is primarily driven by the reaction of metals with atmospheric oxygen and other gaseous compounds, resulting in the formation of oxides or other corrosion products on the metal surface. While generally slower than wet corrosion, dry corrosion can still lead to significant material degradation, particularly in high-temperature conditions where oxidation rates are increased [42], [43].

5.3. Forms of Corrosion:

Corrosion manifests in various forms, including general corrosion [44], which uniformly affects the entire metal surface, often accelerated by oxygen, leading to rust in materials like cast iron and steel. Localised corrosion [44], such as pitting, and crevice corrosion, targets specific areas due to uneven exposure to corrosive fluids, with pitting creating deep holes and crevice corrosion occurring in low-oxygen zones. Stress corrosion cracking (SCC) arises from tensile stress in corrosive environments, while intergranular corrosion affects grain boundaries in stainless steels. Galvanic corrosion occurs when dissimilar metals are electrically connected, causing the more reactive metal to corrode preferentially. Erosion corrosion results from the interaction between fluid velocity and metal surfaces, and waterline corrosion is found in partially submerged metals with varying oxygen levels. Lastly, biological corrosion is driven by microorganisms that alter local chemistry and accelerate corrosion processes. [44], [45], [46].

5.4. Corrosion factors:

The focus of this part is on the different elements that lead to metallic corrosion. It looks at things like temperature, air humidity, pH levels, corrosive product properties, surface film properties, and metal purity. For a thorough analysis of the corrosion process in metals, it is essential to comprehend these elements [44].

5.5. Corrosion in the oil industry:

5.5.1. Pipeline Characteristics:

Pipelines are primarily constructed from low-carbon or low-alloy steel [47], [48] consisting of 98-99% iron with small amounts of carbon and other alloying elements to enhance strength and toughness [47], [48]. They are designed to be durable [48], maintaining their properties under a wide range of temperatures (-20°F to +250°F) [48]. Additionally, elements like chromium, vanadium, niobium, and titanium are incorporated to improve corrosion resistance and mechanical strength [47] [48].

5.5.2. Corrosion in Pipelines:

Pipelines are essential for transporting gases and liquids over long distances, but are highly susceptible to corrosion, which can lead to significant economic losses and environmental damage [49]. Corrosion primarily occurs due to electrochemical reactions in the presence of moisture and corrosive agents, resulting in material degradation [47]. Two main types of corrosion affecting pipelines are external corrosion [50], [48], caused by environmental factors, and internal corrosion [50], [48], which often arises from the materials being transported.

Figure 12: *Corrosion on pipelines.*

5.5.3. Factors Influencing Petroleum Corrosion:

Petroleum corrosion is influenced by several factors that can significantly impact the integrity of pipelines and storage facilities. Key factors include the chemical composition of the transported materials, temperature, pressure, microbial activity (especially from sulphate-reducing bacteria) , material properties and environmental conditions such as humidity and oxygen levels. These elements can accelerate corrosion rates and compromise the structural integrity of pipelines. [47], [49], [51], [52], [53]

5.5.4. Oil Corrosion Control:

To mitigate corrosion in pipelines, various strategies are employed, including careful material selection with carbon steel being the most common choice, alongside alternatives like stainless steel and fibreglass-reinforced plastic. Cathodic protection, which involves applying an electric current to the pipeline and anodic protection, which maintains a passive state, are effective methods. Additionally, protective coatings can be applied and corrosion inhibitors can be added to the oil to prevent corrosion. Biocides may also be utilised to control bacterial growth within the pipeline. [47], [48], [49], [53], [54]

5.6. Corrosion inhibitor:

"A material that, when introduced to an environment in small concentrations, minimises the loss of metal, reduces the extent of hydrogen embrittlement, and protects the metal against pitting" is the definition of a corrosion inhibitor [55].

5.6.1. Features of the inhibitors:

The mechanism of action of corrosion inhibitors is twofold: first, inhibitor molecules are transferred over the metal surface, and then the inhibitor's functional groups interact with the metallic surface. The following crucial factors are taken into account for inhibitors: inhibitor’s solubility in corrosive media. Inhibitor compatibility with corroding systems. Stability of the inhibitor during temperature and pH changes. Cost of an inhibitor. Environmental friendliness. Effectiveness of corrosion inhibition.[56]

5.6.2. Classification of inhibitors:

As a result, several compounds, such as organic molecules, inorganic complexes, natural products, and rare earth elements, have been effectively identified as corrosion-preventing agents for a wide range of metals in various corrosive environments (dry media, molten substances, and aqueous and nonaqueous). Depending on the metals, medium, and environmental factors, each of these inhibitors was shown to be distinct in its activity and efficacy [56].

5.6.3. Domains of Application for Corrosion Inhibitors:

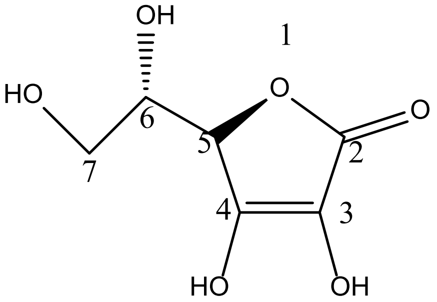
Corrosion inhibitors serve multiple purposes in various applications. In aqueous systems, they reduce corrosion by forming a protective layer or by slowing down electrochemical processes, whether anodic, cathodic, or both. These inhibitors can also be integrated into anodic and conversion coatings, which are active barrier coatings applied industrially, as well as used as pigments in paints [55].

6. Generality about antioxidant potential:

Antioxidants are chemical compounds that can stop other molecules from oxidizing by neutralizing free radicals, unstable species that are naturally produced during cellular metabolism and are distinguished by an absent electron [57]. These radicals, which can be derived from oxygen (ROS) or nitrogen (RNS), cause oxidative stress that damages cells by affecting lipids, proteins, carbohydrates, and nucleic acids. In particular, lipid peroxidation produces toxic compounds like malondialdehyde (MDA) and 4-hydroxynonenal (HNE) [57]. Numerous human diseases, including Alzheimer's, Parkinson's, cancer, atherosclerosis, and even inflammatory and neurodegenerative disorders, are linked to this oxygen-related stress [57], [58]. To stabilize the free radicals, antioxidants often cede an atom of hydrogen to disrupt the chain reactions of oxidation [59]. Factors such as their chemical structure, concentration, distribution within the system, temperature, kind of oxidizable substrate, and the presence of pro-oxidant or synergistic compounds all affect how effective they are [59]. Antioxidant activity refers to the rate at which an antioxidant and an oxidant react, whereas antioxidant capacity quantifies the number of radicals neutralised. According to nutrition and food science, an antioxidant is a chemical that is present at low concentrations and protects biological systems against the harmful effects of reactive species [59], [60]. It does not work on all reductors, just those that have a protective effect [58]. Antioxidants are also used in the agro-food industry to prevent lipid oxidation, which results in undesirable compounds that compromise food safety and quality [61].

6.1. Ascorbic Acid:

Ascorbic acid, often known as vitamin C, is a naturally occurring organic compound that is abundant in many fruits and vegetables (Ascorbic Acid: A Review of its Chemistry and Reactivity). Originally found in the adrenal glands, paprika, oranges, and chou, it was once known as hexuronic acid until its antiscorbic properties led to the term "ascorbic acid" [62]. This crystalline white compound has a molecular mass of 176.1 g/mol and a high fusion point of 193 °C. It is highly soluble in water (33 g/100 mL at 25 °C), forming a colourless solution, but has a much lower solubility in organic solvents such as absolute ethanol (2 g/100 mL), 95% ethanol (3.3 g/100 mL), acetic acid (0.2 g/100 mL), acetonitrile (0.05 g/100 mL), and propylene glycol (5 g/100 mL) [63].
Its molecular structure is characterised by an ethylenediol motif, which is located on carbons C3 and C4 and is essential to its antioxidant activity [63]. As a strong antioxidant and free radical inhibitor, ascorbic acid has a lot of use in the pharmaceutical and nutritional [62]. Notwithstanding its stability when stored in a state of secrecy for several years, ascorbic acid is extremely sensitive to heat, light, oxygen, and pH changes while in aqueous solution. Its oxidation, which changes its biological properties, entails the release of two protons at pH values between 1 and 4 and one proton above pH 5. With dissociation constants $pKa1 = 4.17$ and $pKa2 \approx 11.57$ and an exposure of dissociation speed of around 4.04, its dissociation remains rather constant up to a pH of 8.4 [64]. It is widely used in cosmetics for its anti-ageing properties, but its low stability prevents it from being used more widely [64]. Additionally, high intravenous dosages of ascorbic acid are being investigated as a treatment adjuvant for bacterial and viral infections as well as in several anticancer therapies [65].



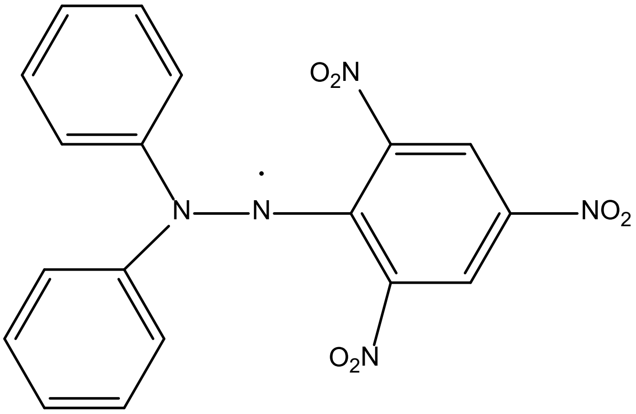
Ascorbic acid

Figure 13: *Chemical structure of ascorbic acid.*

6.2. DPPH:

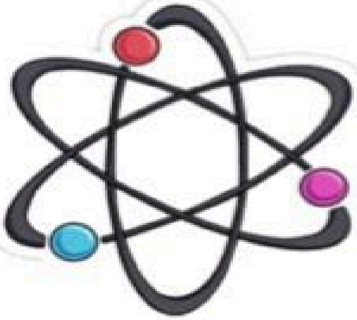
The free radical 1,1-diphenyl-2-picrylhydrazyl (DPPH•) is a stable species whose stability is caused by the single electron's delocalisation across the whole molecule, hence causing its dimerisation. Additionally, this delocalisation gives DPPH• a strong violet colour, with a maximum absorption band about 520 nm that is slightly influenced by the solvent's nature [66], [67], [68]. While it is practically insoluble in water at room temperature, DPPH• is weakly soluble in nonpolar solvents but dissolves well in a variety of polar organic solvents [67], [69]. The absorbance of this radical can be affected by external factors, including light, oxygen, and the pH of the reaction environment [70]. The process of its synthesis usually involves oxidising derivatives of hydrazine in polar solvents like benzene or dichloromethane using strong oxidising agents like lead dioxide, lead tetraacetate, potassium permanganate, or oxidised silver. After easy filtering, these straightforward and efficient reactions allow for the creation of other stable radical hydrazyles that carry carboxyl or sulfonyl groups, and they also enable the extraction of the radical with a nearly quantitative resulting product [67].

The DPPH test is widely used to assess the antioxidative potential of pure compounds or complex extracts, such as those found in fruit, vegetable, or medicinal plant juices [66]. This non-enzymatic method relies on the ability of antioxidants to neutralise the free radical DPPH•, which has a violet colour, by reducing it to DPPH-H, which has a yellow colour, by the transfer of electrons or protons [66]. Spectrophotometry UV-Visible is used to monitor this reaction, with the decrease in absorbance being proportional to the antioxidant activity [67]. This method is easy, quick, reasonable, and reasonably priced, and it allows for the efficient cribbling of many samples in a shorter amount of time [67]. Effectiveness is frequently expressed by the value EC_{50}, which shows the concentration required to reduce 50% of DPPH• radicals. Even though DPPH is not very soluble in water, the test is often conducted using either ethanol or methanol [66], [69].



DPPH

Figure 14: *Chemical structure of DPPH.*



CHAPTER 02:

MATERIALS AND METHODS

1. Turbidimeter:

The analysis of turbidity involves examining the optical characteristics of the water that lead to light absorption and diffusion rather than straight-line transmission. Turbidity manifests as a decrease in water transparency because light deviates when it encounters suspended particles in the water column. Light deviates less from its original path when turbidity is low. Because they can disperse light, particles like limon, clay, algae, organic matter, and microorganisms can be detected. Turbidity is commonly measured with a device called a turbidimeter. To measure low turbidity levels, precise light diffusion detection is required. Today's turbidimeters can detect very low changes in the light intensity through a water sample because of advancements in photodetector technology. [71]

Figure 15: *Turbidimeter.*

2. Jar test:

The first known jar test was conducted in 1918 at the University of California by W.F. Langelier, under the direction of Charles Gilman Hyde, as part of studies on the purification of the city of Sacramento's drinkable water. This experiment aimed to assess the coagulation's effectiveness about various agitation parameters. The device utilized consisted of glass cylinders with rotating poles that allowed for adjustment of the agitation speed. Researchers like Baylis, Peterson, Fleming, and Bartow have since improved this technique by creating various agitator designs, whether they are rotative or vertically moving. Additionally, their work has shed light on the factors that determine pH, agitation speed, coagulant type, and the ionic composition of water. They also established a distinction between the conditioning phase, which corresponds to slow flocculation, and the rapid mixing phase, which aims to homogenize the reactive ones. As time went on, the jar test became a crucial and widely used tool for studying and optimizing coagulation-flocculation processes in water treatment. [72]

Figure 16: *Jar test.*

3. Differential Scanning Calorimetry (DSC):

Differential scanning calorimetry (DSC) measures the thermal exchanges related to chemical reactions or physical transitions of a material. It works by comparing the heat flow between a sample and a reference under a controlled temperature program. When the sample changes, a difference in thermal flux is recorded and converted into a differential signal. This technique makes it possible to determine the amount of heat released, either wholly or partially, the calorific capacity, the transition temperatures, and other characteristics like crystallinity or purity. Due to its extreme precision, DSC has become a vital tool for thermal analysis of materials. [73]

4. Thermogravimetric Analysis (TGA):

The thermogravimetric analysis uses a very accurate balance and a four-point scale in which the temperature increases linearly with time. These days, it's common practice to continuously and automatically record temperature and weight. This results in a thermogravimetric curve that shows how weight decreases with temperature (or time). The results can also be shown as a thermogravimetric differential curve, which shows the rate of weight loss with temperature. These curves help to identify elements like plateaus, inflexions, and changes in the rate of weight loss, giving important information on thermal stability and subsequent reactions. The thermogravimetric analysis is very helpful for determining the process temperatures of decomposition and assessing thermal stability. [74]

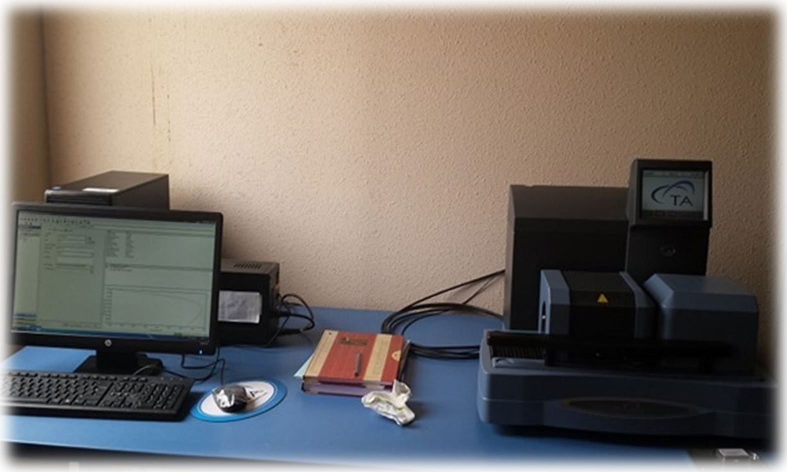


Figure 17: *Thermogravimetric Analyzer*.

5. Ultraviolet–Visible Spectrophotometry (UV–Vis):

UV-VIS spectroscopy is a physical optical analysis method that uses light in the visible and ultraviolet. It is based on the Beer-Lambert law, which states that a solution's absorbance is proportional to both the duration of the light's path and the concentration of the absorbent species. The concentration of an absorbent compound in a solution can therefore be ascertained using this constant length. It is essential to comprehend how absorbance changes with concentration to make a reliable interpretation. The basic idea behind it is that when molecules or ions absorb light, an electronic transition occurs inside their structure. Utilized for over three decades, this approach has established itself as a leading analytical tool in modern laboratories due to its ease of use, versatility, speed, accuracy, and low cost. [75]

Figure 18: *UV-VIS* Spectrophotometer.

6. Infrared Spectroscopy (IR):

IR spectroscopy quantifies how much infrared light is absorbed by a material's chemical bonds. Functional groups, which are chemical structural fragments of molecules, tend to absorb infrared radiation in the same frequency range, independent of the structure of the molecule in which they are located. It is possible to identify unknown molecules' structures and track their structural or chemical changes thanks to the link between a molecule's structure and the frequencies at which it absorbs infrared light. [76]

Figure 19: *Infrared Spectroscopy (IR).*

7. X-ray diffraction (XRD):

Max von Laue and his colleagues demonstrated in 1912 that crystalline solids could diffract X-rays, which had a wavelength equivalent to the distance between an atom's atomic planes. This discovery marked the beginning of X-ray diffraction (XRD), which is now widely used to analyse the crystal structure of materials. This approach, which is based on the Bragg law ($n\lambda = 2d\sin\theta$), relies on the constructive interference of monochromatic X-rays directed towards a crystal sample. The X-rays, which are produced in a tube with cathodic rays, are filtered, concentrated, and then directed toward the sample; a detector then records the diffracted rays. The process of powder diffraction makes it possible to assess their purity, characterize minerals or clays, measure mesh parameters, and examine the texture of minced films. The non-destructive, precise, and multivalent method of X-ray diffraction has become a crucial tool for structural analysis in many scientific and industrial fields. [77]

Figure 20: *X-ray diffractor.*

8. The potentiometer (pH):

The pH is a crucial parameter that allows one to assess how acidic or basic an aqueous environment is, where the majority of natural reactions occur. Theoretically, it is defined as the logarithm decimal negative of the activity of hydrogen ions (aH^+), or $pH = -\log_{10}(aH^+)$. This definition was introduced by Danish chemist Sørensen in 1909 and updated in 1924 to take into account electrochemical measurement techniques. This activity is frequently associated with the concentration of ions H^+ in very diluted solutions. For instance, pure water at 25 °C with an autoprotolysis constant of 10^{-14} has 10^{-7} mol/L of H^+ and OH^- ions, which is equivalent to a pH of 7. A pH below 7 indicates an acidic environment, whereas a pH over 7 indicates a basic environment. In practice, pH is measured using electrodes sensitive to H+ ions, whose potential complies with the Nernst law since it depends on the ion activity. The reference method uses a liquid electrochemical cell without a junction made up of an Ag/AgCl reference electrode and a hydrogen electrode ($Pt/H_2/H^+$). The pH of an unknown solution is then determined by comparing the measured potential difference (emf) between the sample to be analysed and a known pH solution. [78]

Figure 21: *pH-meter.*

9. Gravimetric:

The gravimetric method is a traditional experimental approach used to study metal corrosion and assess inhibitor effectiveness. It aims to precisely quantify the mass loss of a metal exposed to a corrosive environment, such as a 1 M solution of hydrochloric acid (HCl), which replicates aggressive industrial settings. Its principle is based on the rigorous testing of metal objects, such as carbon steel plates, both before and after they are submerged in a corrosive solution for a certain amount of time. The mass loss is used to determine the rate of corrosion after the samples have been cleaned and dried. By repeating the experiment with varying inhibitor doses, the effectiveness of the inhibitors may be calculated by calculating the inhibition rate, surface coverage (θ), and corrosion rate reduction. Despite being based on straightforward measurements, this approach offers a straightforward, repeatable, and dependable assessment of the inhibitors' anticorrosion behaviour. It also makes it possible to evaluate different chemical families, such as non-ionic surfactants, by demonstrating their ability to adsorb to metal surfaces and generate protective layers that inhibit the effect of acidic environments. [79]

Figure 22: *The gravimetric method.*

10. Operating procedure:

Product κ-CG-g-PAM (1):

In a 150 mL flask, 1 g of carrageenan was dissolved in 40 mL of hot water at 70°C. Simultaneously, in a separate beaker, 0.5 g of acrylamide was dissolved in 3 mL of water, and 0.1 g of APS was dissolved in 2 mL of distilled water in a hemolysis tube. Once the carrageenan was completely dissolved, the acrylamide solution was added to the carrageenan solution. The mixture was then stirred for 15 minutes, while maintaining a temperature of 70°C. The APS solution was then poured onto the mixture of carrageenan and acrylamide. The reaction was run under reflux for 1 hour at 70°C. Finally, the product was precipitated by adding ethanol, then washed with hot water.

Product κ-CG-g-PAM (2):

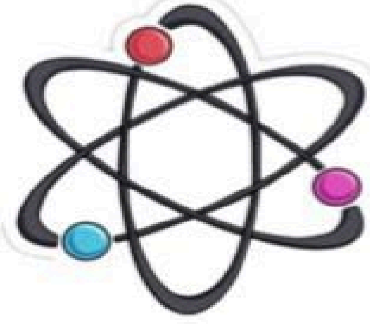
In a 150 mL flask, 1 g of carrageenan was dissolved in 40 mL of hot water at 70°C. At the same time, 2 g of acrylamide was dissolved in 3 mL of water in a beaker. Once the carrageenan was completely dissolved, 0.1 g APS was dissolved in 2 mL distilled water in a haemolysis tube. Once the APS had dissolved, it was added to the carrageenan solution and left to stir for 15 minutes, maintaining the temperature at 70°C. The acrylamide solution was then poured over the mixture of carrageenan and APS. The reaction was run under reflux for 1 hour at a temperature of 70°C. Finally, the product was precipitated with ethanol and washed with hot water.

Product κ-CG-g-PAM (2'):

In a 150 mL flask, 1 g of carrageenan was dissolved in 40 mL of hot water at 70°C. At the same time, 1 g of acrylamide was dissolved in 3 mL of water in a beaker. Once the carrageenan was completely dissolved, 0.1 g APS was dissolved in 2 mL distilled water in a haemolysis tube. Once the APS had dissolved, it was added to the carrageenan solution and left to stir for 15 minutes, maintaining the temperature at 70°C. The acrylamide solution was then poured over the mixture of carrageenan and APS. The reaction was run under reflux for 1 hour at a temperature of 70°C. Finally, the product was precipitated with ethanol and washed with hot water.

Product κ-CG-g-PAM (2"):

In a 150 mL flask, 1 g of carrageenan was dissolved in 40 mL of hot water at 70°C. At the same time, 0.5 g of acrylamide was dissolved in 3 mL of water in a beaker. Once the carrageenan was completely dissolved, 0.1 g APS was dissolved in 2 mL distilled water in a haemolysis tube. Once the APS had dissolved, it was added to the carrageenan solution and left to stir for 15 minutes, maintaining the temperature at 70°C. The acrylamide solution was then poured over the mixture of carrageenan and APS. The reaction was run under reflux for 1 hour at a temperature of 70°C. Finally, the product was precipitated with ethanol and washed with hot water.



CHAPTER 03:
RESULTS AND DISCUSSION

1. Synthesis of copolymers:

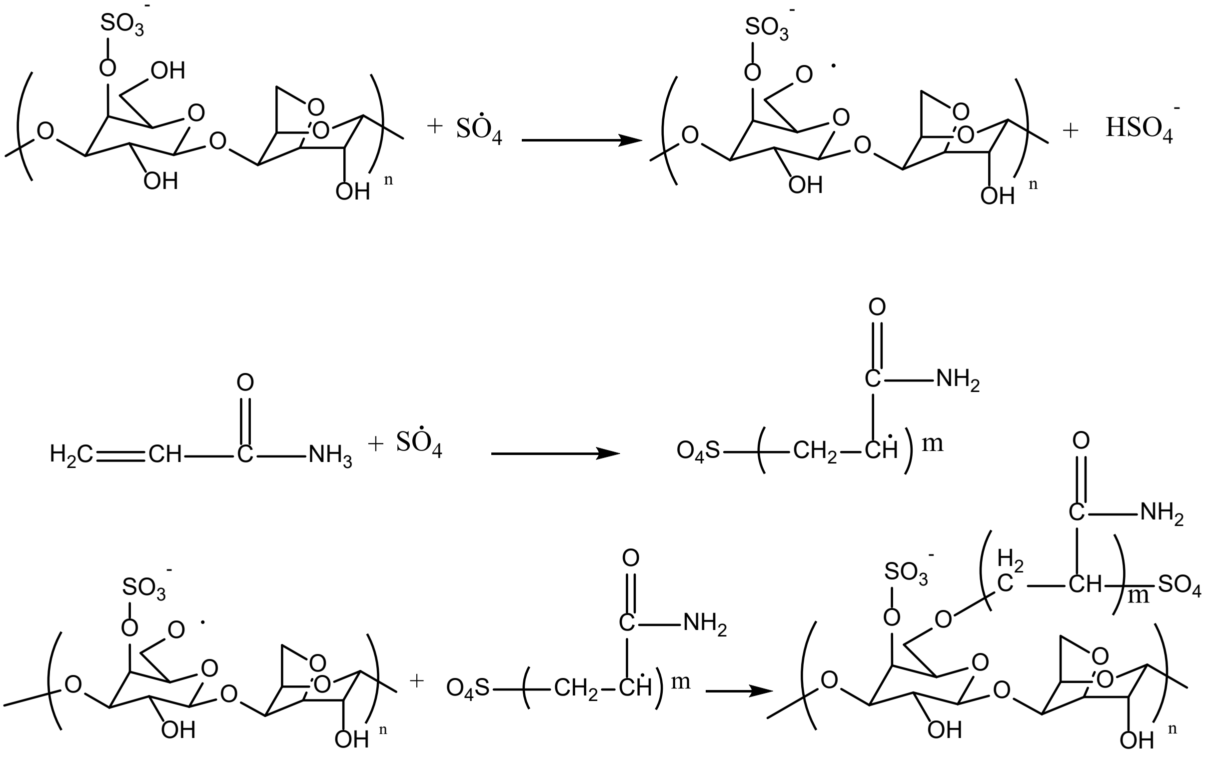
Table 1: *κ-CG-g-PAM Synthesis: Variable Factors and Shared Protocol.*

Product	Acrylamide (g)	Addition Order	Common Conditions (All Products)
κ-CG-g-PAM (1)	0.5	κ-CG → AM → APS	• 1 g of κ-CG dissolved in hot water at 70°C. • 0.1 g of APS solution. • Acrylamide dissolved in water separately. • Stirring for 15 min at 70°C. • Reflux for 1 hour at 70°C. • Precipitation with ethanol. • Washing with hot water.
κ-CG-g-PAM (2)	2	κ-CG → APS → AM	
κ-CG-g-PAM (2′)	1	κ-CG → APS → AM	
κ-CG-g-PAM (2″)	0.5	κ-CG → APS → AM	

2. Copolymerization reactions:

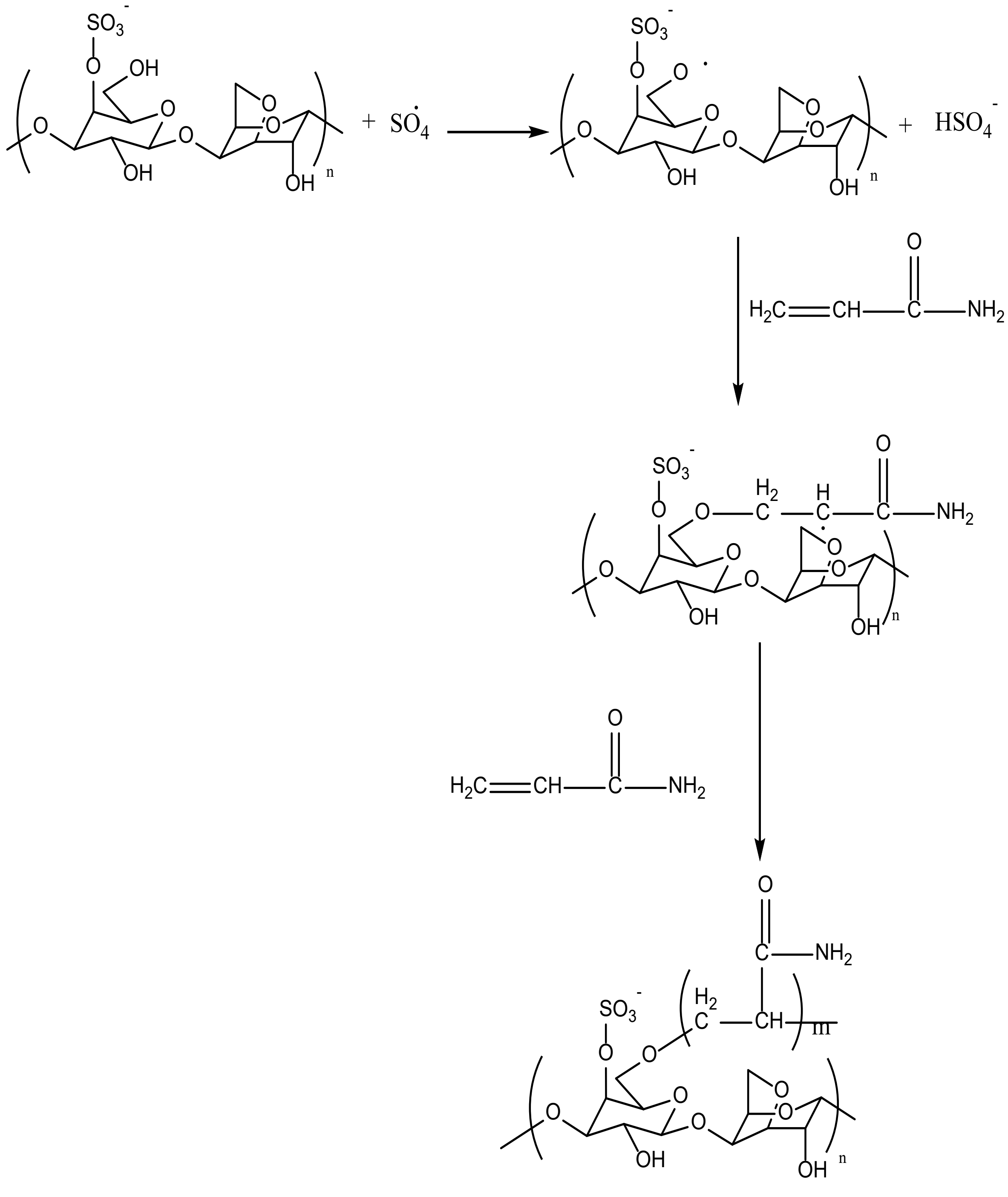
- **Copolymerization reaction of κ-CG-g-PAM (1):**

$$S_2O_8^{2-} \xrightarrow[T\ (60°C)]{} 2S\dot{O}_4 + HSO_4^-$$



- **Copolymerization reaction of κ-CG-g-PAM (2), κ-CG-g-PAM (2'), and κ-CG-g-PAM (2"):**

$$S_2O_8^{2-} \xrightarrow[\text{T (60°C)}]{} 2\dot{S}O_4 + HSO_4^{-}$$



3. Characterisation of copolymers:

3.1. Infrared Spectroscopy (IR):

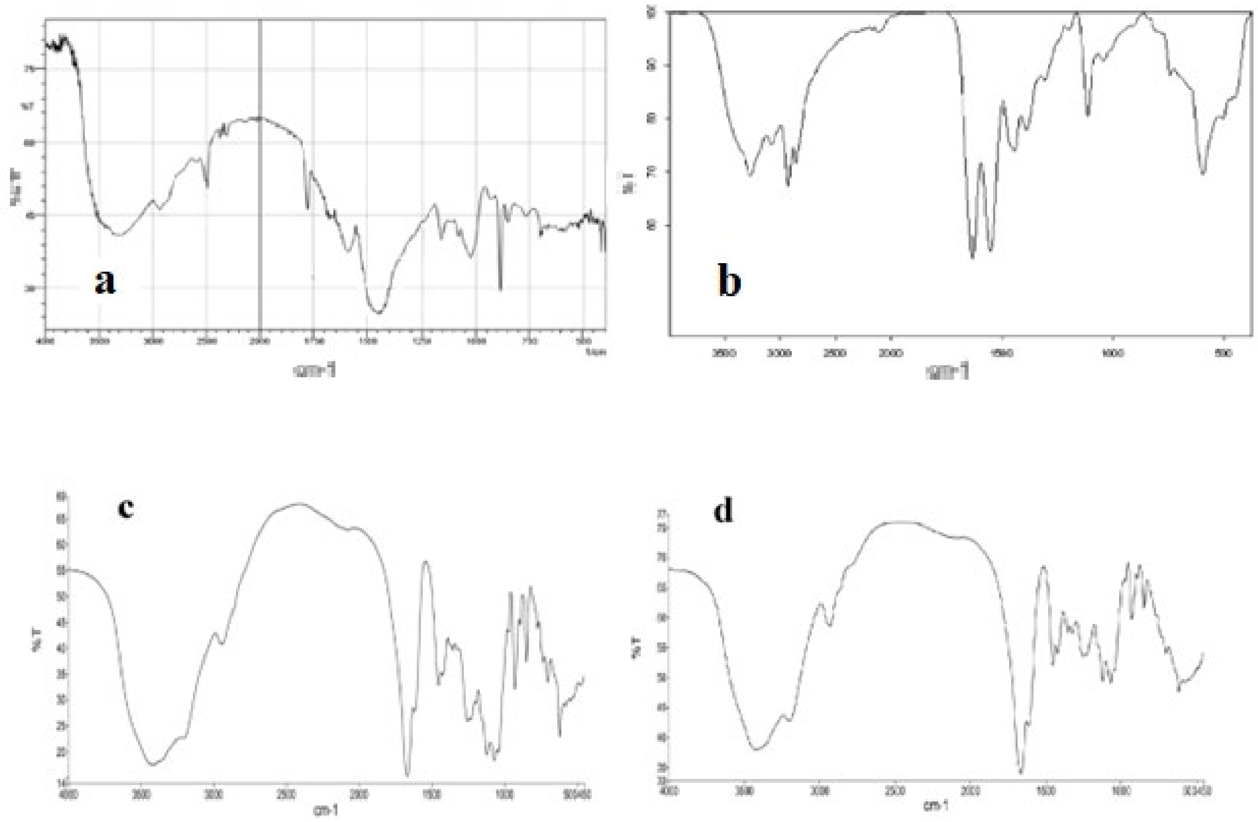


Figure 23: *FTIR spectra of: a) kappa-carrageenan (κ-CG), b) acrylamide (AM), c) κ-CG-g-PAM (1), and d) κ-CG-g-PAM (2).*

The spectra in **Figure 23. a**, **23.c**, and **23.d** show an absorption band at 1669 cm−1, corresponding to C=O stretching, and another at 1454 cm−1 for C-N stretching, indicating the presence of acrylamide functional groups. A distinctive feature of kappa-type carrageenan (κ-CG), **Figures 23.b**, **23**.**c**, and **23.d,** is an absorption band at 1069 cm−1, indicating the presence of a glycosidic bond. Additionally, a band at 928 cm−1 is associated with 3,6-anhydrogalactose, while a band at 847 cm−1 relates to the galactose-4-sulfate group. Both acrylamide and κ-CG are represented in the functional groups of the κ-CG-g-PAM copolymer. Grafting occurred on the -OH group rather than the sulphate moiety, as indicated by a less intense band at 3415 cm−1 attributable to -OH groups, which is weaker than the parent κ-CG [80].

3.2. Thermogravimetric Analysis (TGA):

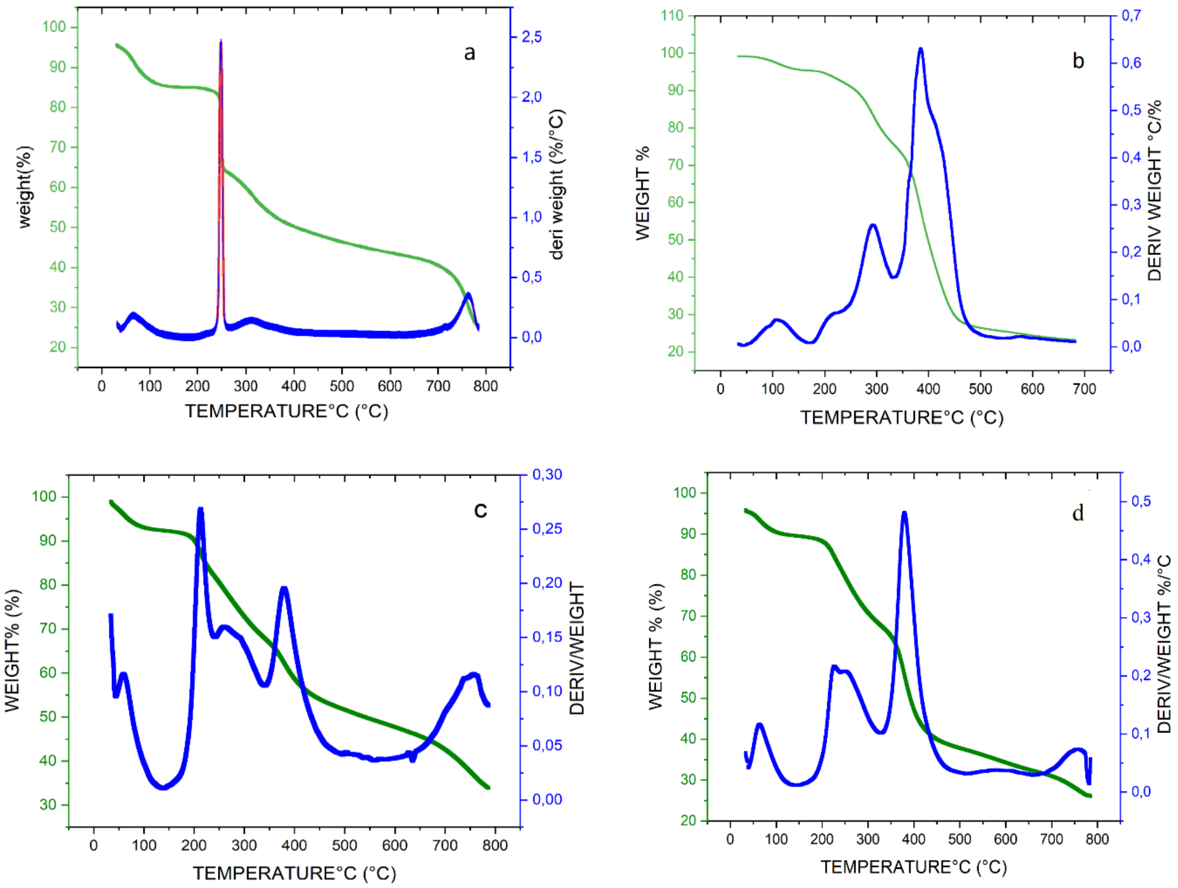


Figure 24: *TGA and DTG curves of: a) kappa-carrageenan, b) PAM, c) κ-CG-g-PAM (1), and d) κ-CG-g-PAM (2).*

Table 2: *Allocation of κ-CG, PAM, κ-CG-g-PAM (1), and κ-CG-g-PAM (2).*

	Dehydration	**Decomposition of κ-CG**	**Decomposition of PAM**	**% Modification rate**
	T<150 °c % Weight loss	150°c<T<240°C % Weight loss	240°c<T<500 % Weight loss	/
κCG	10.58	59.33	/	
PAM	5	/	73	
Co (κ-CG-PAM) (1)	6.63	10.69	30.01	73.73
Co (κ-CG-PAM) (2)	5.93	7.85	43.85	84.82

Based on this table, it is observed that in the case of κ-CG and PAM, two mass losses occur: the first, at a temperature below 150 °C, is due to the evaporation of absorbed water, while the second results from the decomposition of the polymers, occurring between 150 and 240 °C for κCG and between 240 and 500 °C for PAM. As for the copolymers, four distinct mass losses are identified: the first, below 150 °C, corresponds to the removal of absorbed water; the second, between 150 and 240 °C, is attributed to the thermal decomposition of carrageenan; the third, between 240 and 500 °C, is related to the degradation of polyacrylamide; and the fourth, above 500 °C, corresponds to the formation of carbonaceous residues.

3.3. Differential Scanning Calorimetry (DSC):

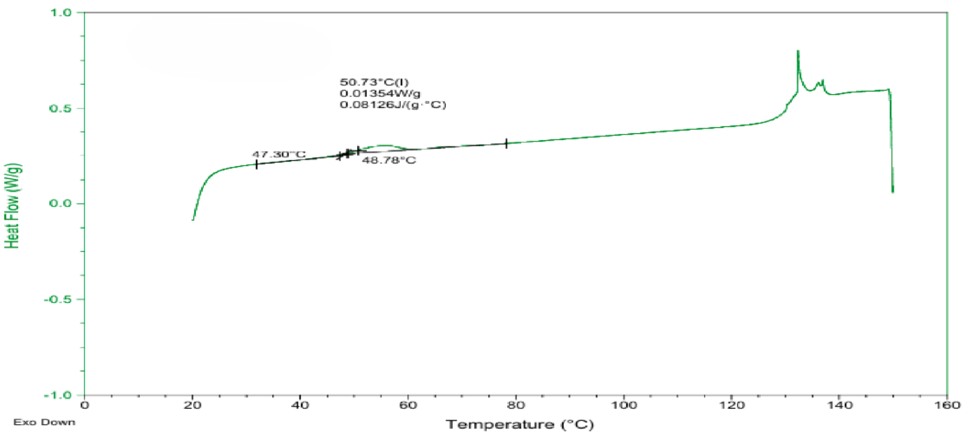


Figure 25: *DSC thermogram of κ-CG-g-PAM (1).*

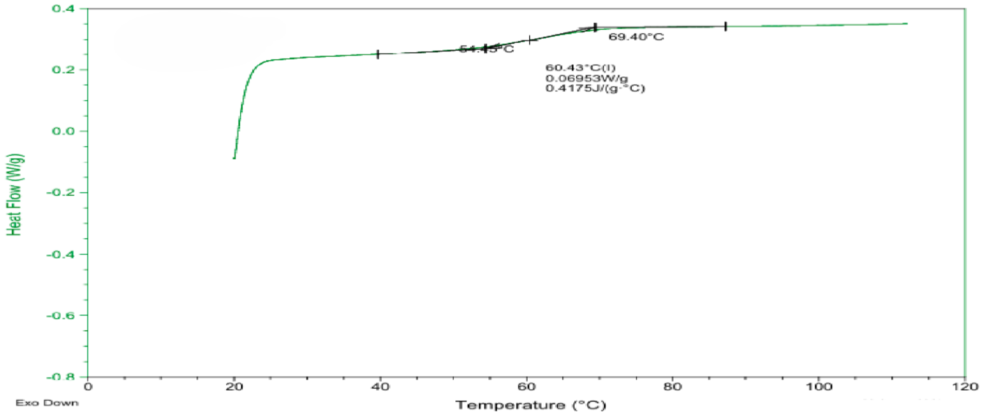


Figure 26: *DSC thermogram of κ-CG-g-PAM (2).*

The two thermograms analyse the heat flow as a function of temperature for the copolymers co(κ-CG-g-PAM) (1) and κ-CG-g-PAM (2), respectively. The first thermogram, **Figure 25,** reveals two transitions: a glass transition at 48.78°C and a melting temperature (Tm) at 132.36°C, characteristic of a semi-crystalline structure. In contrast, the second thermogram, **Figure 26,** shows a single thermal transition, corresponding to a glass transition temperature (Tg) of 54.45 °C, indicating that the copolymer has an amorphous structure. This difference in thermal behaviour can be explained by the nature of the starting polymers, namely κ-carrageenan (κ-CG) and polyacrylamide (PAM), which are often semi-crystalline polymers. κ-CG has a Tg of approximately 41.59 °C and a Tm of 85.77 °C [81] while PAM has a Tg of 161.4 °C and a Tm of 237 °C [82]. The amorphous structure observed in copolymer (2) may be due to the difficulty in organizing macroradicals during polymerization, limiting the formation of crystalline regions. In contrast, copolymer (1), with better molecular organization, retains a certain degree of crystallinity, which explains its semi-crystalline nature. These results highlight the influence of synthesis conditions on the final structure of the material [83].

3.4.X-ray diffraction (XRD):

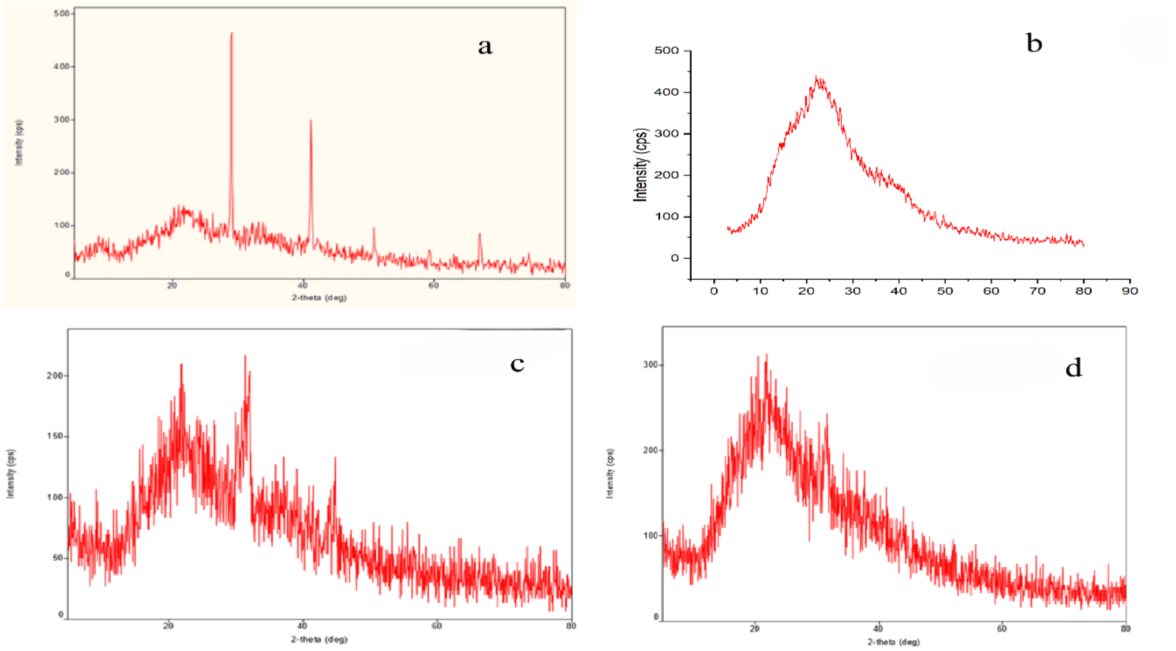


Figure 27*: XRD pattern of: a) kappa-carrageenan, b) PAM, c) κ-CG-g-PAM (1), and κ-CG-g-PAM (2).*

The nature of pure kappa-carrageenan is semicrystalline, as shown in **Figure 27. a**. In contrast, **Figure 27. b** presents the XRD spectrum of PAM, which indicates an amorphous morphology [84]. **Figure 27. c** depicts the XRD of copolymer 1, which shows an amorphous structure with some crystalline regions, as previously demonstrated by DSC. Similarly, Figure d indicates an amorphous structure for copolymer 2.

The distinctive XRD profile, characterized by a prominent halo and the absence of well-defined peaks, is typical of amorphous or low-crystalline materials. In copolymers derived from grafted polysaccharides, such as kappa-carrageenan grafted with polyacrylamide, the grafting process disrupts the regular arrangement of polymer chains, preventing the formation of an ordered crystalline network. As a result, the structure exhibits amorphous characteristics, which are represented by a diffractive halo in the XRD diffractogram [85].

κ-CG-g-PAM (1) exhibits lower solubility compared to κ-CG-g-PAM (2). Consequently, our research concentrated on the coagulation and flocculation, as well as the corrosion resistance and antioxidant characteristics of κ-CG-g-PAM (2).

4. Coagulation and flocculation:

4.1.Natural settling of bentonite:

The bentonite naturally settles to an initial concentration of 100 mg/L with an initial turbidity value of 26.25 NTU before turbidity is removed from bentonite suspensions by coagulation and flocculation. The necessary amount of BN is dissolved in distilled water to create this concentration, which is then allowed to settle without being stirred. After a week of settling, the residual turbidity was measured as a function of time. Table 2 displays the findings.

The following formula is used to determine the removal rate.

$$\text{Removing turbidity (\%)} = \left(\frac{TU_0 - TU}{TU_0}\right) \times 100$$

Therefore:

TU_0: Turbidity of the initial bentonite suspension (NTU).

TU: Residual turbidity of bentonite suspension (NTU).

Table 3: *Observation of the natural sedimentation of bentonite suspension at 100 mg/L at various times.*

Settling time (hours)	1	3	5	24	72	93	145	166
Elimination rate (%)	11.34	15.68	18.00	35.03	45.08	48.73	50.90	53.00
Turbidity residual (NTU)	23.27	22.13	21.53	17.06	14.42	13.46	12.89	12.34

The results of the table are shown in Figure 28.

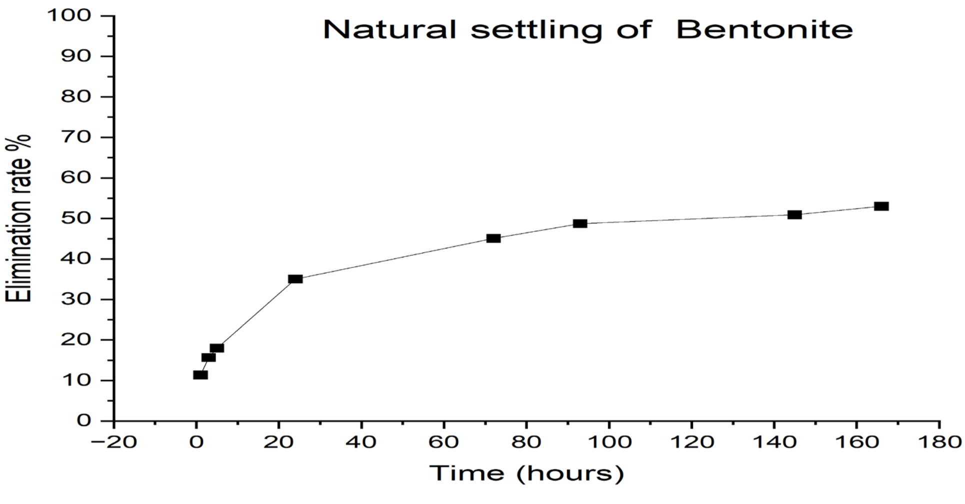


Figure 28: *Natural settling of Bentonite 100 mg/L.*

The findings indicate that bentonite settles naturally at a maximum rate of 53%, which is sluggish [86]. The bentonite's crystalline structure, surface charge, and composition could all contribute to this poor elimination rate. Surface charge and stability behaviour are related, and surface charge varies with pH. The protonation of the bentonite's hydroxyl groups (Al-OH, Si-OH) results in a positively charged surface at low pH. The hydroxyl groups on the surface experience deportation and an inversion of the positive to negative charges when the pH rises.

4.2.Coagulation/flocculation of turbid bentonite suspensions:

A technique called coagulation-flocculation, which involves the injection of copolymer solutions and is followed by a Jar test device, was used to increase the removal rate of bentonite suspensions. The following is the experimental protocol:

Preparation of copolymer solutions κ-CG-PAM (2), κ-CG-PAM (2'), and κ-CG-PAM (2'') at a concentration of 1 g/L at 20°C and neutral pH is prepared. Making bentonite suspensions at 20°C and neutral pH with a concentration of 100 mg/L (100 ppm). Put 250 mL of a 100 ppm bentonite solution into each beaker, and if needed, use HCl or NaOH (1M) to bring the pH down. To bracket the ideal concentration, introduce the coagulant volumes κ-CG-PAM (2); κ-CG-PAM (2'); κ-CG-PAM (2''). After placing the beakers in the Jar test apparatus, begin agitation at 150 rpm for 5 minutes. Then, reduce the speed to 50 rpm for 7 minutes. After this process, measure the turbidity of small samples of the supernatant at sedimentation times of 5, 10, and 15 minutes.

Table 4: *The optimal concentrations of flocculant for the three copolymers κ-CG-g-PAM (2), κ -CG-g-PAM (2'), and κ-CG-g-PAM (2'') (settling time 5 minutes) in bentonite suspension at 100 mg/L.*

Flocculant	Optimum flocculant concentration (g/L)	pH	Removed turbidity (%)
Copolymer κ-CG-g-PAM (2)	0.0157 g/L	pH= 3,26	31.92
		pH=7,03	24.72
		pH=8,61	31.62
Copolymer κ-CG-g-PAM (2′)	0.0016 g/L	pH=2,26	45.49
		pH=7	43.96
		pH=8,89	38.63
Copolymer κ-CG-g-PAM (2″)	0.0079 g/L	pH=2,62	35.43
		pH=7,45	41.94
		pH=9,7	39.62

4.3.Natural settling of kaolin:

The natural settling of kaolin was monitored for an initial concentration of 100 mg/L with an initial turbidity value equal to 71 NTU. This concentration is prepared by dispersing the necessary quantity of Kaolin in distilled water and leaving it to settle without stirring. The residual turbidity was measured as a function of time during one week of settling. The results are shown in Table 4.

Table 5: *The natural settling of kaolin suspension at a concentration of 100 mg/L over time.*

Settling time (hours)	1	3	5	24	51	126	145	169
Elimination rate (%)	15.14	31.20	38.08	61.43	75.60	85.71	86.93	88.31
Turbidity residual (NTU)	60.25	48.85	43.96	27.39	17.32	10.15	9.28	8.30

The table's results are illustrated in Figure 29.

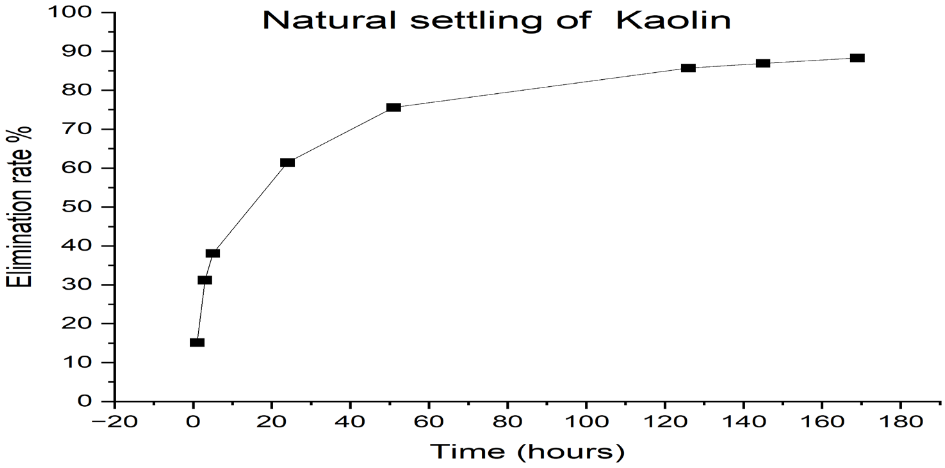


Figure 29: *Natural settling of Kaolin 100 mg/L.*

According to this figure, the rate of sedimentation of the kaolin suspension (100 mg/L) increases over time, peaking at 88.31% after one week of sedimentation. These results are notably higher compared to the natural sedimentation of bentonite. This can be attributed to a significant number of suspended particles that aggregate and settle quickly. Consequently, the water with a higher turbidity level was the easiest to sediment naturally.

4.4.Coagulation/flocculation of turbid kaolin suspensions:

The following is the experimental protocol:

Preparation of copolymer solutions κ-CG-PAM (2), κ-CG-PAM (2'), and κ-CG-PAM (2") at a concentration of 1 g/L at 20°C and neutral pH is prepared. Making kaolin suspensions at 20°C and neutral pH with a concentration of 100 mg/L (100 ppm). Put 250 mL of a 100 ppm kaolin solution into each beaker, and if needed, use HCl or NaOH (1M) to bring the pH down. To bracket the ideal concentration, introduce the coagulant volumes κ-CG-PAM (2); κ-CG-PAM (2'); κ-CG-PAM (2"). After placing the beakers in the Jar test apparatus, begin agitation at 150 rpm for 5 minutes.

Then, reduce the speed to 50 rpm for 7 minutes. After this process, measure the turbidity of small samples of the supernatant at sedimentation times of 5, 10, and 15 minutes.

Table 6: *The optimal concentrations of flocculant for the three copolymers κ -CG-g-PAM (2), κ -CG-g-PAM (2'), and κ-CG-g-PAM (2") (settling time 5 minutes) in kaolin suspension at 100 mg/L.*

Flocculant	Optimum flocculant concentration (g/L)	pH	Removed turbidity (%)
Copolymer κ-CG-g-PAM (2)	0.0196 g/L	pH= 2,46	23.94
		pH= 6.97	43.17
		pH= 9.66	-07.04
Copolymer κ-CG-g-PAM (2’)	0.0079 g/L	pH=2,52	21.13
		pH=5,96	31.75
		pH=9,9	-23.94
Copolymer κ-CG-g-PAM (2’’)	0.0119 g/L	pH=2,25	38.80
		pH=7,06	54.97
		pH=8,86	-14.09

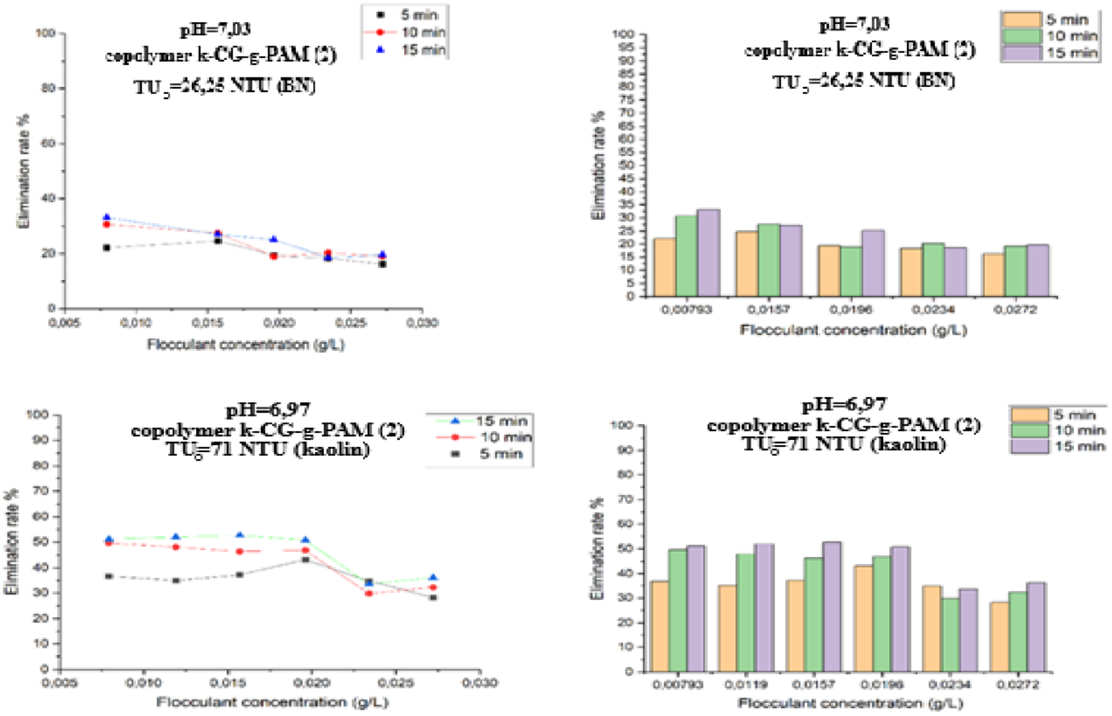


Figure 30: *The optimal concentrations of flocculant for κ -CG-g-PAM (2)*

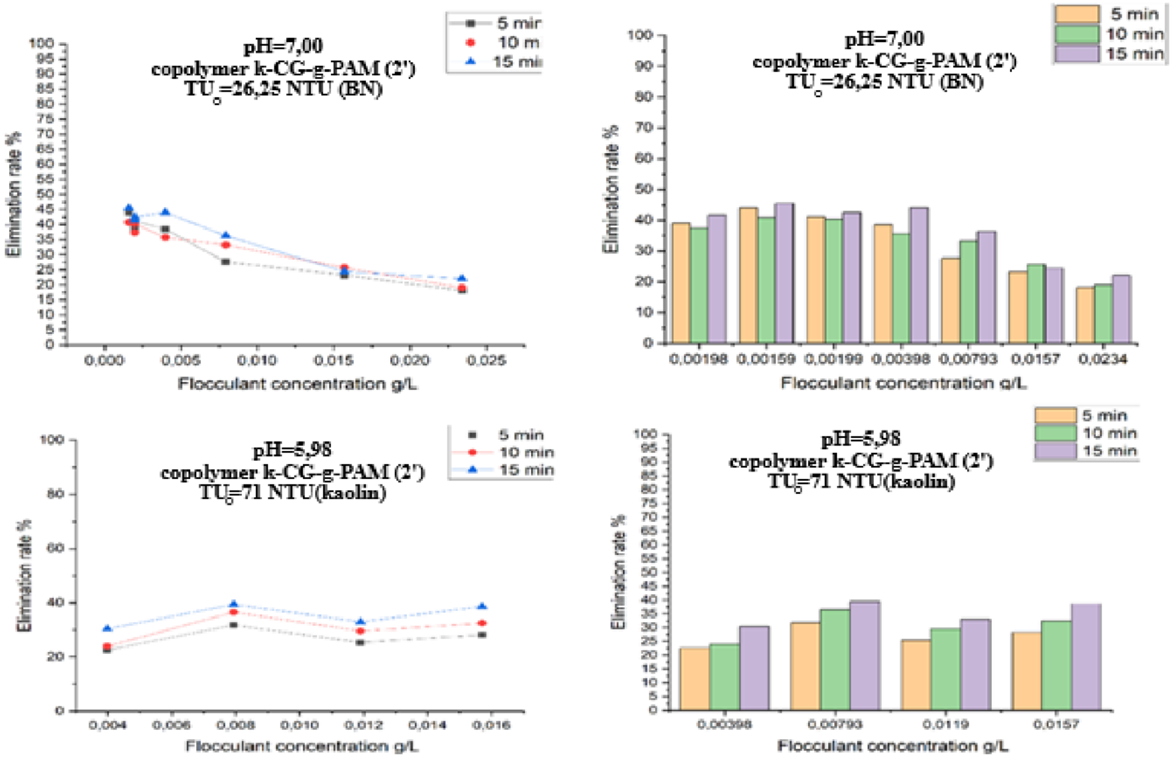


Figure 31: *The optimal concentrations of flocculant for κ -CG-g-PAM (2').*

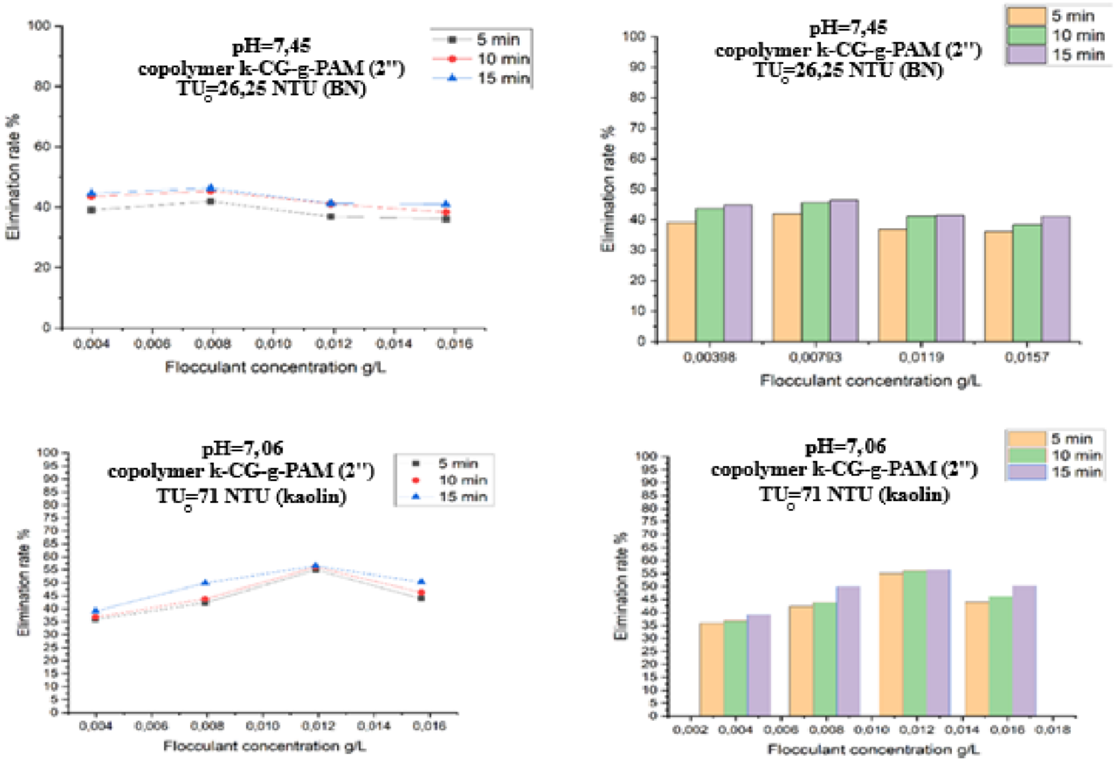


Figure 32: *The optimal concentrations of flocculant for κ -CG-g-PAM (2'').*

The copolymers κ-CG-g-PAM (2), κ-CG-g-PAM (2'), and κ-CG-g-PAM (2") demonstrate different optimal concentrations for removing turbidity from a 100 mg/L bentonite suspension. Specifically, κ-CG-g-PAM (2) is effective at a concentration of 0.0157 g/L, achieving a removal rate of 24.72%. In comparison, κ-CG-g-PAM (2') attains a removal rate of 43.96% at 0.00159 g/L, while κ-CG-g-PAM (2") shows a removal rate of 41.94% at 0.00793 g/L.

For a 100 mg/L kaolin suspension, the κ-CG-g-PAM (2) copolymer removes 43.17% of turbidity at a concentration of 0.0196 g/L. κ-CG-g-PAM (2') is effective at 0.00793 g/L with a removal rate of 31.75%. κ-CG-g-PAM (2") achieves a removal rate of 54.97% at a concentration of 0.0119 g/L. All removal rates were measured after 5 minutes of decantation.

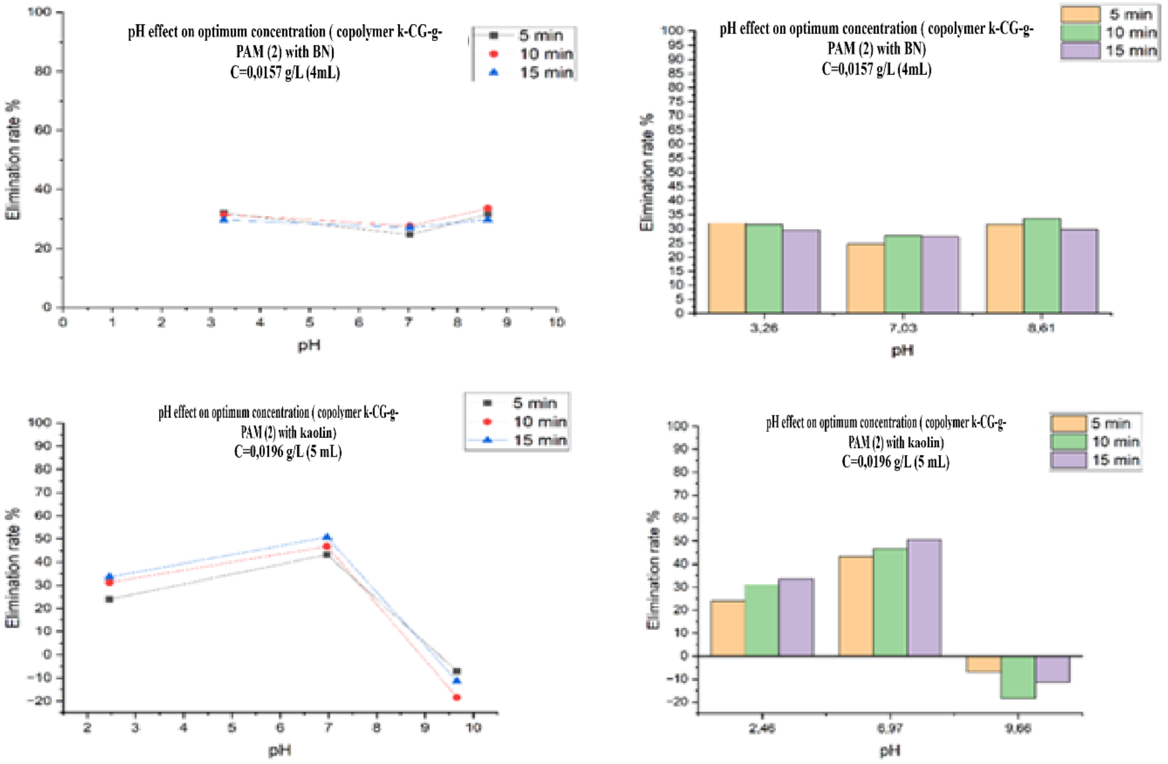


Figure 33: *The pH effect on optimum concentrations of κ -CG-g-PAM (2).*

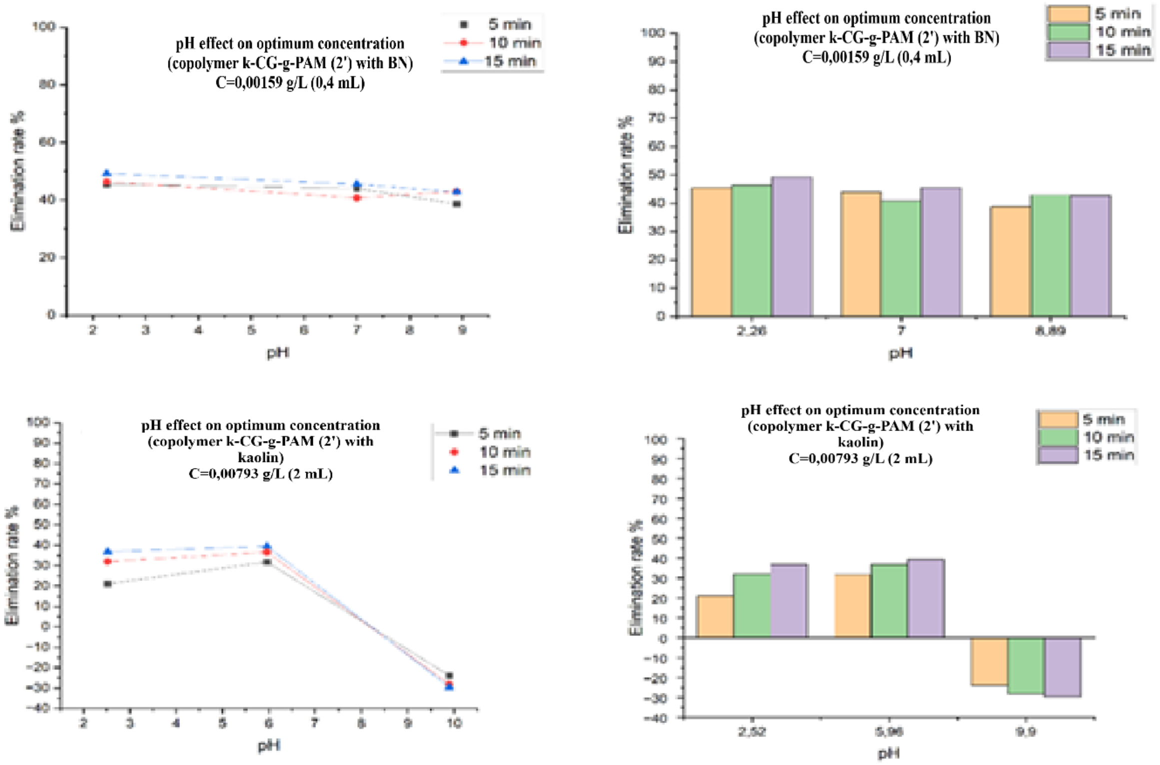


Figure 34: *The pH effect on optimum concentrations of κ -CG-g-PAM (2').*

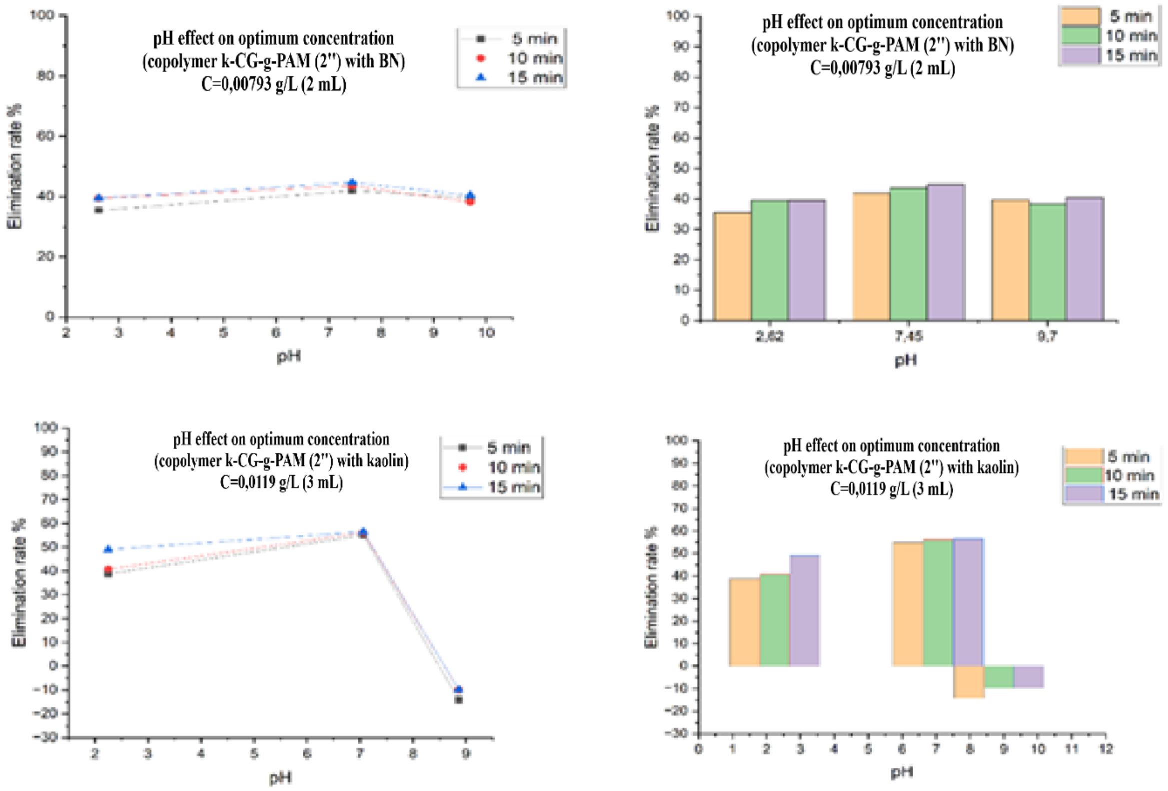


Figure 35: *The pH effect on optimum concentrations of κ -CG-g-PAM (2'').*

Figures **33**, **34**, and **35** illustrate the effect of pH on the optimal concentration of κ -CG-g-PAM (2), κ -CG-g-PAM (2'), and κ -CG-g-PAM (2") in suspensions of BN (100 mg/L) and kaolin (100 mg/L).

The pH significantly influences the percentage of turbidity removal for the κ -CG-g-PAM (2) copolymer in the BN suspension. The removal percentage increases in acidic (approximately 31.92%) and basic (31.62%) environments compared to neutral conditions (24.72%). For the κ -CG-g-PAM (2') and κ -CG-g-PAM (2") copolymers, the observations vary:

κ -CG-g-PAM (2'): The percentage of turbidity removal increases in an acidic environment, reaching 45.49%. However, it decreases in a basic environment (38.63%) compared to a neutral environment (43.96%).

κ -CG-g-PAM (2"): The percentage of turbidity removal decreases in both acidic (35.43%) and basic (39.62%) environments compared to a neutral environment (41.94%).

In the case of the kaolin suspension, the percentage of turbidity removal generally decreases in both acidic and basic conditions compared to neutral conditions:

In an acidic environment:

κ -CG-g-PAM (2): 23.94%

κ -CG-g-PAM (2'): 21.13%

κ -CG-g-PAM (2"): 38.80%

In a basic environment:

κ -CG-g-PAM (2): -7.04%

κ -CG-g-PAM (2'): -23.94%

κ -CG-g-PAM (2"): -14.09%

The elimination percentages in a neutral environment for the kaolin suspension are 43.17% for κ -CG-g-PAM (2), 31.60% for κ -CG-g-PAM (2'), and 54.97% for κ -CG-g-PAM (2").

The results can be understood by considering the following explanations:

The ability of a copolymer to interact with colloids that lead to turbidity decreases when it is poorly soluble in the water being treated. Furthermore, for effective particle aggregation, synthetic polymers such as acrylamide require a high molecular weight, typically in the range of 6 to 8 × 10^6. Improper grafting can alter this structure.

Coagulation-flocculation processes depend on the neutralization of colloid charges. Anionic carrageenans, such as kappa, may not adequately counteract the negative charges of suspended particles due to several factors: [87]

- ✓ Electrostatic Repulsion: The negative charges of both carrageenans and suspended particles repel each other, which prevents the formation of flocs.[87]

- ✓ Formation of Unstable Complexes: The interactions between carrageenans and particles may not be strong enough to create stable flocs, leading to inefficient particle separation. [87]

The double network system involves the formation of two interconnected networks: the natural network of carrageenan (a sulphated polysaccharide) and the synthetic network formed by the polymerisation of grafted acrylamide. This structure gives the copolymer high mechanical strength and a high retention capacity for colloidal and suspended particles, promoting the formation of stable, voluminous flocs that facilitate decantation or filtration. [88]

In an acidic environment ($pH < 4$), the amine groups of acrylamides become protonated (NH_3^+), resulting in the copolymer having a partial positive charge. This charge offsets the negative charges of the bentonite/kaolin particles (generally negatively charged), promoting aggregation by electrostatic neutralisation. [89]

The flocculation efficiency of kappa carrageenan grafted acrylamide copolymers is significantly affected by the pH of the solution. In acidic conditions, the copolymer exhibits more cationic sites, which enhances its electrostatic attraction to negatively charged particles like bentonite and kaolin, leading to improved turbidity removal. However, when NaOH is added, the pH increases, making the solution more basic. This change reduces the positive charge density on the copolymer chains, weakening the electrostatic interactions necessary for effective flocculation and resulting in decreased removal efficiency [90]. The effect of adding NaOH can vary depending on the specific structure of the graft copolymer, its molecular weight, and the amount used. Generally, graft copolymers with higher molecular weights perform better in removing turbidity. However, their effectiveness can still be negatively impacted at high pH levels for the reasons mentioned above. [91]

The results show that our copolymers are effective but not excellent flocculants for reducing turbidity, which aligns with the results presented by BELAID Abdelaali [92].

5. Corrosion:

The X60 steel is a pipeline steel, typical for corrosion resistance in oil/gas environments [93].

Table 7: *The chemical composition of X60 steel* [93].

Element	**C**	**Si**	**Mn**	**P**	**S**	**Cu**	**Mo, Ni**	**Cr, V**	**Fe**
Composition (wt.%)	0.20	0.36	1.16	0.01	<0.003	0.18	0.10	0.08	Balance

Measurement of corrosion speed:

The following law determines the value of corrosion speed:

$$W_{corr} = \frac{\Delta m}{S \times t}\ (m.h^{-1}.cm^{-2})$$

With:

Δm: Loss of mass (mg)

S: the surface of the sample in cm^2

t: the time of immersion (h)

Inhibitor efficacy:

The effectiveness inhibitor percentage (E%) of the compounds under study is determined using the following relation:

$$E\% = \frac{W_{corr} - W_{corr\,(inh)}}{W_{corr}} \times 100$$

With:

W_{corr} and $W_{corr(inh)}$ denote mass loss without inhibitor and with inhibitor.

Table 8: *Corrosion speed and inhibition efficiency from gravimetric measurements on X60 steel after 2 hours of immersion at 25 °C in a 1M HCl solution in the presence of the copolymers κ-CG-g-PAM (2), κ-CG-g-PAM (2'), and κ-CG-g-PAM (2'') at various concentrations.*

	C (g/L)	W_{corr} ($mg.h^{-1}.cm^{-2}$)	E%
Bare steel	/	0.48	/
κ-CG-PAM (2)	0.1	0.32	32.23
	0.3	0.32	31.96
	0.6	0.33	30.76
κ-CG-PAM (2′)	0.1	0.44	08.17
	0.3	0.44	06.74
	0.6	0.34	28.36
κCG-PAM (2″)	0.1	0.37	22.11
	0.3	0.32	33.65
	0.6	0.25	47.84

This table presents the results of the corrosion speed and inhibition efficiency obtained from gravimetric measurements on X60 steel after 2 hours of immersion at 25 °C in a 1M HCl solution, both in the absence and in the presence of the copolymers κ-CG-g-PAM (2), κ-CG-g-PAM (2′), and κ-CG-g-PAM (2″) at various concentrations.

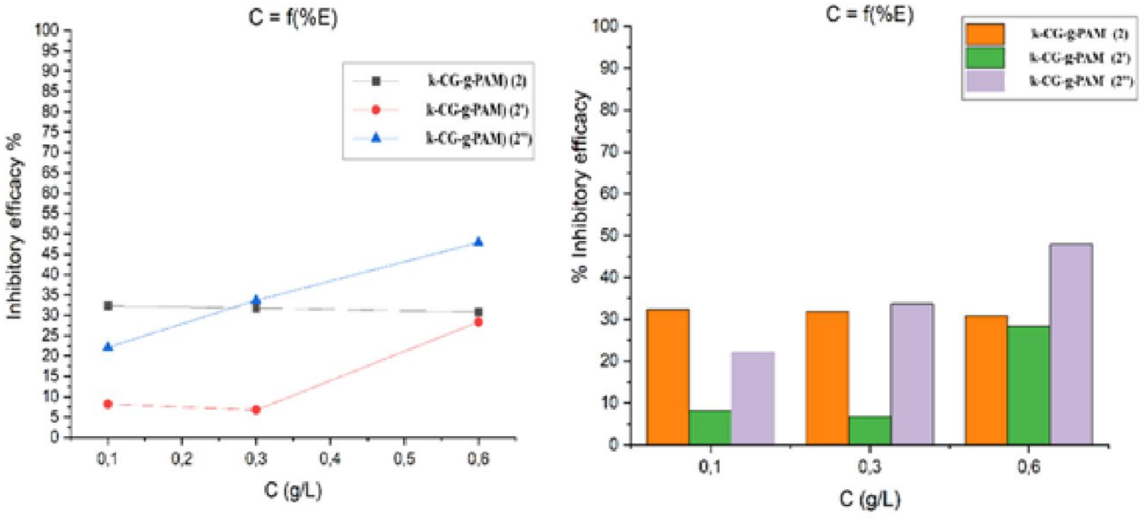


Figure 36: *The variation in the inhibitory efficiency percentage of Co(κ-CG-g-PAM) (2), Co(κ-CG-g-PAM) (2'), and Co(κ-CG-g-PAM) (2") in 1M HCl.*

The two graphs illustrate the variation in the inhibitory efficiency percentage of the copolymers κ-CG-g-PAM (2), κ-CG-g-PAM (2′), and κ-CG-g-PAM (2″) as a function of their concentration in a corrosive medium (1M HCl). It is observed that the inhibitory efficiency of κ-CG-g-PAM (2) remains stable at around 32%. In contrast, the efficiency of κ-CG-g-PAM (2′) increases slightly, reaching 8.17% at a concentration of 0.1 g/L, then decreases to 6.74% at 0.3 g/L, before rising again to 28.36% at 0.6 g/L. As for κ-CG-g-PAM (2″), its inhibitory efficiency increases steadily with concentration. These results can be attributed to the degradation of κ-carrageenan (κ-CG) in an acidic medium (HCl), a phenomenon confirmed by the study entitled "The influence of the conformational state of κ- and ι-carrageenan on the rate of acid hydrolysis" [94]. This suggests that our copolymers are poor corrosion inhibitors.

The results indicate that our copolymers are ineffective in preventing corrosion of mild steel in acidic environments, which aligns with the results presented by HAMZAOUI et al [95].

6. Antioxidant potential:

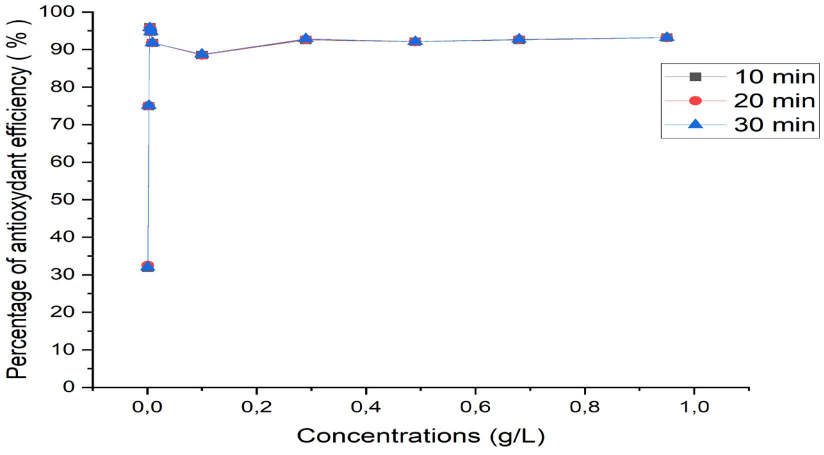


Figure 37: *The percentage of antioxident efficiency of ascorbic acid at different times.*

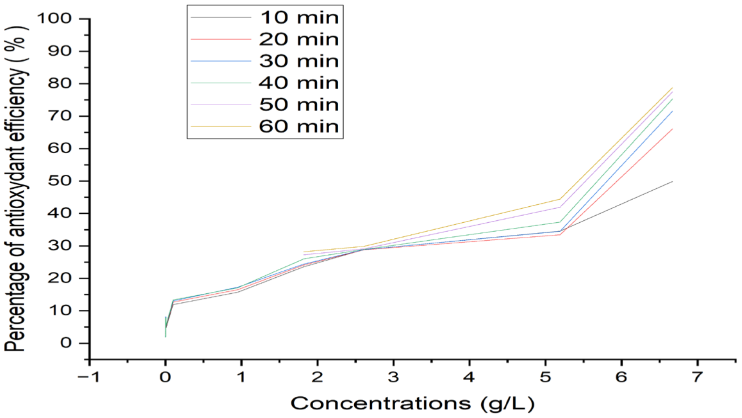


Figure 38: *The percentage of antioxidant efficiency of κ-CG-g-PAM (2") at different times.*

One of the graphs illustrates the percentage of antioxidant efficiency of ascorbic acid as a function of its concentration at different time intervals (**Figure 37)**. At the same time, the other shows the same for the copolymer kappa-carrageenan grafted with acrylamide κ-CG-g-PAM (2") (**Figure 38)**. In the case of ascorbic acid, the curves corresponding to different times overlap, and its antioxidant efficiency increases with concentration, up to 0.29 g/L, beyond which the efficiency plateaus, indicating a saturation in its ability to scavenge DPPH free radicals. For the copolymer, all curves show a general increase in antioxidant power with concentration, a typical behaviour for antioxidants but with variations depending on time: the curves corresponding to different reaction times (from 10 to 60 minutes) exhibit varying efficiency levels at the same concentration, suggesting that experimental conditions significantly influence antioxidant activity. At low concentrations, the increase in antioxidant activity over time is minimal, possibly due to the limited amount of active copolymer. At intermediate concentrations, the effect of time becomes more pronounced, indicating a stronger influence of reaction time on efficiency. At high concentrations (≥ 2.61 g/L), the antioxidant efficiency is already relatively high even at shorter times and tends to reach a plateau over longer durations, suggesting saturation of active sites or complete reaction with DPPH.

To determine the EC50 value, we identify the section of the graph that represents the effective concentration corresponding to 50 % antioxidant efficiency.

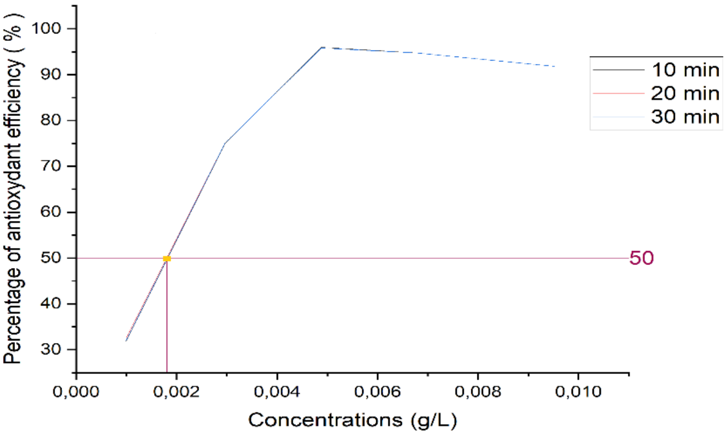


Figure 39: *The EC50 of ascorbic acid at 10 minutes.*

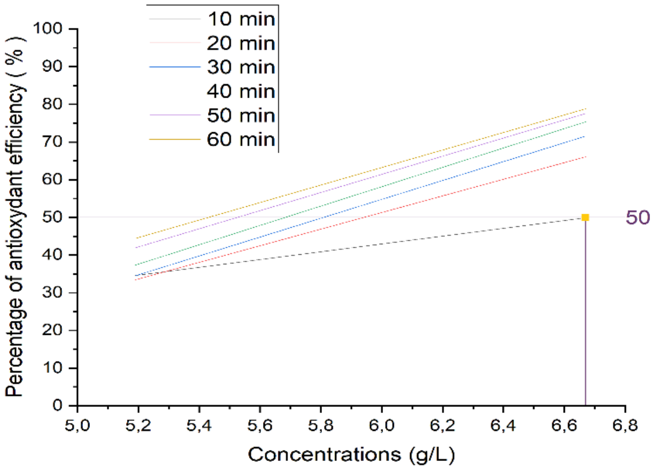
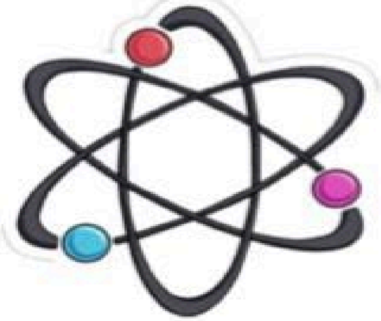


Figure 40: *The EC50 of κ-CG-g-PAM (2") at 10 minutes.*

Extrapolation of the curves at 50% antioxidant efficiency allowed us to determine the following EC_{50} values: 1.82×10^{-3} g/L for ascorbic acid and 6.68 g/L for our copolymer. The lower EC_{50} value of ascorbic acid indicates a higher antioxidant activity compared to the copolymer [96]. This difference can be explained by the ability of ascorbic acid to release two protons at a pH between 1 and 4 (pH=2.93) [97], enhancing its reducing power, whereas our copolymer releases only one proton.
Furthermore, a comparison between the EC_{50} value of our copolymer (6.68 g/L) and that of pure κ-carrageenan (2.65 g/L) also reveals a decrease in antioxidant activity following copolymerization. This suggests that the modified structure of the copolymer may alter the functional groups involved in antioxidant activity [7].



GENERAL CONCLUSION

Our study successfully modified kappa carrageenan (κ-CG) through a grafting process with acrylamide (AM), effectively incorporating acrylamide functional groups primarily onto the hydroxyl groups of κ-CG. This modification was confirmed using various characterization methods. Infrared spectroscopy (IR) and thermogravimetric analysis (TGA) demonstrated the successful modification. Both differential scanning calorimetry (DSC) and X-ray diffraction (XRD) indicated an amorphous structure for the copolymers, with XRD revealing an amorphous profile that lacked defined crystalline peaks. These spectroscopic and thermal analyses collectively validate the effectiveness of our synthesis method, as well as the structural and functional changes in the copolymer.

A significant advantage of the synthesized copolymer is its ability to dissolve in cold water, which is highly beneficial for various aqueous formulations. In terms of functional properties, the copolymers, specifically κ-CG-g-PAM, exhibited considerable antioxidant potential, with efficacy increasing in relation to concentration until reaching a plateau. This characteristic makes them promising materials for antioxidant applications, particularly in the food and pharmaceutical sectors. However, their performance in other areas was more limited. The coagulation-flocculation efficiency was moderate, with turbidity removal rates ranging from 24.72% to 43.96% for bentonite and up to 54.97% for kaolin, depending on pH and flocculant concentration. Additionally, their effectiveness as corrosion inhibitors in acidic media (1M HCl) was low, with inhibition levels ranging from 6.74% to 47.84%, suggesting limited usefulness in this field.

In conclusion, while the modification of κ-CG with acrylamide leads to copolymers with diverse structures, their most notable performance lies in their antioxidant capabilities. However, their coagulation-flocculation and corrosion inhibition functionalities indicate that further adjustments, along with alternative formulations or modifications, are needed to optimize their performance and maximize their potential in various industrial applications.

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ملخص:

تناقش هذه المذكرة تعديل الكابا كاراجينان (κ-CG) من خلال إضافة مادة الأكريلاميد (AM) لتحسين خواصه لاستخدامه في معالجة المياه ومنع التآكل وكمضاد للأكسدة. تتضمن هذه الدراسة مراجعة شاملة للأدبيات المتعلقة بالكاراجينان، وتفاصيل عن مصادره وأنواعه، بالإضافة إلى توصيف البوليمرات المشتركة المُصنَّعة باستخدام تقنيات تحليلية مختلفة، مثل التحليل الطيفي بالأشعة تحت الحمراء، والتحليل الحراري الوزني، والأشعة السينية. تشير النتائج إلى أن البوليمرات المشتركة المعدلة تُظهر نشاطًا كبيرًا مضادًا للأكسدة، مع زيادة الفعالية جنبًا إلى جنب مع التركيز. ومع ذلك، كانت كفاءة التخثر والتلبد لمعلقات البنتونيت والكاولين معتدلة، وكانت قدرتها على تثبيط التآكل في الأوساط الحمضية منخفضة. تشير النتائج إلى أنه على الرغم من أن التعديل يعزز بعض الخصائص الوظيفية، إلا أن هناك حاجة إلى مزيد من التعديل لتحسين أدائها في التطبيقات الصناعية.

الكلمات المفتاحية: الكابا كاراجينان، الأكريلاميد، معالجة المياه، التخثر والتلبد، منع التآكل، مضاد للأكسدة.

Abstract:

This memoir discusses the modification of kappa carrageenan (κ-CG) through grafting with acrylamide (AM) to improve its properties for applications in water treatment, corrosion prevention, and as an antioxidant. The study includes a thorough literature review on carrageenan, detailing its sources and types, as well as the characterization of the synthesized copolymers using various analytical techniques, such as infrared spectroscopy, thermogravimetric analysis, and X-ray diffraction. The results indicate that the modified copolymers demonstrate significant antioxidant activity, with efficacy increasing alongside concentration. However, their coagulation-flocculation efficiency for bentonite and kaolin suspensions was moderate, and their ability to inhibit corrosion in acidic media was low. The findings suggest that while the modification enhances certain functional properties, further optimization is needed to improve their performance in industrial applications.

Key words: kappa-carrageenan, acrylamide, water treatment, coagulation and flocculation, corrosion inhibition, antioxidant potential.

Résumé :

Ce mémoire traite de la modification du carraghénane kappa (κ-CG) par greffage avec l'acrylamide (AM) afin d'améliorer ses propriétés pour des applications dans le traitement de l'eau, la prévention de la corrosion et en tant qu'antioxydant. L'étude comprend un examen approfondi de la littérature sur le carraghénane, détaillant ses sources et ses types, ainsi que la caractérisation des copolymères synthétisés à l'aide de diverses techniques analytiques, telles que la spectroscopie infrarouge, l'analyse thermogravimétrique et la diffraction des rayons X. Les résultats indiquent que les copolymères modifiés sont plus résistants que les carraghénanes traditionnels. Les résultats indiquent que les copolymères modifiés présentent une activité antioxydante significative, dont l'efficacité augmente avec la concentration. Cependant, leur efficacité de coagulation-floculation pour les suspensions de bentonite et de kaolin était modérée, et leur capacité à inhiber la corrosion en milieu acide était faible. Les résultats suggèrent que si la modification améliore certaines propriétés fonctionnelles, une optimisation plus poussée est nécessaire pour améliorer leurs performances dans les applications industrielles.

Mots clés : kappa-carraghénane, acrylamide, traitement de l'eau, coagulation et floculation, inhibition de la corrosion, potentiel antioxydant.