



Separation of Crude Oil and Its Derivatives Spilled In Seawater by Using

Commiphora Myrrha and Coal Fly Ash

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ABSTRACT

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This investigation delved into the comprehensive assessment of a composite amalgam featuring hybrid nanoparticles composed of CoFe_2O_4 , an extract from Commiphora Myrrha, and Coal Fly Ash. The primary objective of this study was to ascertain the potency of this composite in addressing the intricate challenge of marine oil spill mitigation, particularly within the context of pervasive environmental contamination. The synthesis of the hybrid nanoparticles was executed at Sebha University, with the remaining components being procured from established commercial sources. The meticulous characterization of these constituents was conducted employing advanced analytical techniques, namely X-ray diffraction spectroscopy (XRD) and infrared absorption spectroscopy (FTIR). The empirical findings gleaned from the experimental investigation conspicuously spotlight the superior efficacy achieved through the strategic employment of hybrid NPs, specifically the CoFe_2O_4 nanoparticles combined with Commiphora Myrrha extract, at a compositional ratio of 20:80. Under these conditions, an appreciable oil removal efficiency of 30.7% was realized, demonstrating the pronounced influence of the amalgamation on the mitigation process. Notably, this efficacy was realized at a concentration of 0.06 g, underscoring the optimal operational parameter within this experimental framework. Remarkably, the pinnacle of success was encountered when hybrid NPs were synergistically combined with coal fly ash, configuring a proportion of 30:70. This synergistic alliance yielded a conspicuously impressive oil removal efficiency of 69.7% under identical concentration conditions of 0.06 g. This outcome significantly underscores the strategic potential of the composite in the context of marine oil spill remediation. The ramifications of these findings are notable, as they contribute to the advancement of knowledge in the realm of environmental contamination management. Furthermore, the demonstrated efficacy of this composite prompts further inquiry into refining and optimizing the proportionality of its constituents and their application methodologies. It is recommended that subsequent studies delve into the mechanisms underlying this composite's performance enhancement, paving the way for the potential integration of this innovation into practical strategies aimed at alleviating the detrimental effects of marine oil spills on aquatic ecosystems.

Keywords: Nanoparticles; Commiphora Myrrha; Coal fly ash; Oil spills removal; waste materials; Hybrid Nanoparticles.

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1. INTRODUCTION

Accidental oil spills stemming from incidents involving the dispersion of petroleum substances, often originating from maritime vessel mishaps, have been recognized as a recurring environmental challenge, predominantly affecting marine environments. Noteworthy instances of such ecological perturbations include the al-Kuwait oil conflagration of 1991, wherein a substantial volume of oil was discharged into seawaters [1,2]. Similarly, the Deep-water Horizon catastrophe in 2010, situated within the expansive expanse of the Gulf of Mexico,

precipitated the release of an estimated 206 million gallons of petroleum derivatives into the aquatic milieu [3-5]. These incidents, emblematic of a broader pattern, underscore the critical need for enhancing and optimizing the efficacy of oil spill remediation methodologies; given that global cumulative oil spillage has reached staggering proportions, exceeding 400,000 tons [6,7].

These incidents not only pose immediate threats to marine life but also pose long-term ecological and socio-economic consequences. The dispersal of oil in water bodies engenders deleterious impacts

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on aquatic ecosystems, manifesting as habitat degradation, toxicological ramifications, and disruptions in the food chain [8,9]. Furthermore, the encroachment of oil slicks onto coastlines can adversely impact local economies, ranging from tourism and fisheries to shoreline real estate. Hence, the imperative to evolve and augment the methodologies employed for oil spill mitigation is underscored by both environmental and economic considerations [10].

One of the key challenges in seawater pollution is the high viscosity and strong adhesion properties of crude oil, which can lead to significant contamination of adsorbents, separation materials, or even their deactivation. In recent years, superhydrophilic materials have been designed based on a combination of their surface morphology and affinity with water to effectively separate oil from water while possessing excellent oil-repellent properties [11–13]. However, most of these materials are capable of only repelling light oils or model oils and are still susceptible to contamination by crude oil residues. Their superior water absorption and retention capabilities distinguish them from other hydrophilic materials.

Among these materials, Nano cellulose extracted from natural plant cell walls has garnered significant attention. The outstanding oil-repellent property of Nano cellulose arises from its unique chemical composition, in which hydrophilic groups are closely and densely linked to the surface of the nanomaterials. This intrinsic property facilitates efficient oil-water separation. The development and utilization of these materials are pivotal in addressing the complex challenges associated with marine oil pollution. The multifaceted interactions between oil and water, compounded by the intricate physicochemical attributes of the marine environment, necessitate innovative and adaptive solutions. In light of these considerations, Nano cellulose emerges as a promising candidate for mitigating oil spills and reducing their detrimental effects [14–17].

Also, the utilization of Commiphora Myrrha [18] in the context of oil separation from seawater represents a notable advancement in environmental remediation strategies. Commiphora Myrrha, a natural material with hydrophilic and adsorptive characteristics, has demonstrated considerable potential as an adsorbent for oil removal due to its inherent affinity for hydrophobic substances. The resin's complex composition, including terpenoids, phenolic compounds, and polysaccharides, contributes to its efficient oil-absorbing properties [19]. Through a combination of physical interactions, such as adsorption and cohesion, Commiphora Myrrha can effectively capture and encapsulate oil molecules, resulting in the separation of crude oil from the aqueous environment. Moreover, its biodegradability and eco-friendly nature make it a compelling candidate for sustainable oil spill mitigation efforts. The application of Commiphora Myrrha in oil separation signifies a harmonious integration of natural materials and scientific innovation, presenting a viable pathway toward addressing the challenges posed by oil contamination in marine ecosystems [20–22].

Efforts geared towards the amelioration of oil spillage necessitate a holistic approach, encompassing the development of innovative technologies for containment, recovery, and remediation. Novel materials and strategies, such as the hybrid nanoparticles comprised of CoFe_2O_4 , Commiphora Myrrha, and Coal Fly Ash explored in this study, stand as emblematic examples of contemporary endeavors aimed at addressing this multifaceted predicament [20]. By deepening our comprehension of the intricate physicochemical interplays dictating the behavior of oil-water interfaces and capitalizing on the advancements in novel materials, the feasibility arises to conceptualize and implement interventions characterized by markedly enhanced efficacy in the purification of oil-contaminated aqueous ecosystems. Such advancements stem from a comprehensive grasp of the intermolecular forces, adsorption phenomena, and molecular arrangements that underlie these interfaces [23–25]. Leveraging cutting-edge materials, with tailored properties and functionalities, enables the strategic design of sorbents, dispersants, and emulsifiers that optimize the separation, dispersion, and degradation of oil pollutants. These interventions, rooted in scientific insights, pave the way for more efficient and environmentally responsible approaches to tackling the persistent challenge of oil contamination in aquatic environments [26–28].

Moreover, the convergence of fundamental understanding and innovative materials holds the potential to drive the development of sustainable technologies with a broader spectrum of applications, extending beyond oil spill remediation to encompass diverse environmental and industrial contexts.

By synergistically combining hybrid nanoparticles composed of CoFe_2O_4 , Commiphora Myrrha extract, and Coal Fly Ash into a nanostructured composite, a novel avenue emerges for addressing marine oil spills. This innovative approach capitalizes on the distinctive properties of each constituent to achieve efficient oil-water separation. The utilization of such a composite holds promise as a potent solution for mitigating the intricate challenges posed by oil leakage in marine environments. The integration of CoFe_2O_4 nanoparticles serves to enhance the separation process through their responsive magnetic properties, allowing for facilitated manipulation and recovery of the composite from the aqueous medium. Commiphora Myrrha extract, characterized by its hydrophilic and adsorptive attributes, contributes to the composite's oil-absorbing capacity. Furthermore, the inclusion of Coal Fly Ash, renowned for its adsorption and affinity for hydrophobic substances, synergistically augments the composite's overall oil removal efficiency.

In consideration of the aforementioned attributes delineating the distinctive qualities of spinel magnetic nanoparticles, rendering them highly auspicious within the realm of water technology for diverse contaminant species, the present investigation is directed towards the attainment of the following objectives:

Investigation of the feasibility of the employment of a hybrid absorbent composite (incorporating spinel magnetic nanoparticles in conjunction with Commiphora Myrrha and spinel magnetic nanoparticles in tandem with coal ash), with the principal objective of effecting the elimination of crude oil and its derivatives from aqueous mediums.

Attainment of a heightened echelon of oil adsorption efficacy through the utilization of a novel and economically viable absorbent material characterized by diminished cost and augmented operational efficiency.

2. Experimental Part

The oil removal tests were performed as described previously in the literature [29–31]. In this test (30 ml) of seawater, 1 ml of crude oil was injected, nanomaterial was added with the addition of, and then it was weighed and left for 30 minutes:

$$\text{GOR} = \frac{\text{weight of baker with sample (Oil + Powder + Water)} - \text{weight of baker after construction}}{\text{total weight of Powder + Oil}}$$

$$\text{Oil removal percentage} = (1 - \text{GOR}) \times 100$$

Where GOR is Gravimetric Oil Removal



Figure 1. Process of crude oil removal from seawater using Nano magnetite (a) mixture of crude oil with CFA-CFO in seawater, (b) attraction of crude oil with CFA-CFO in seawater by magnetite.

3. Results and discussion

3.1. Chemical composition of Commiphora Myrrha (CM), Cobalt Ferrite Oxide (CFO) and Coal fly ash (CFA)

Table 1 presents a comprehensive depiction of the chemical constituents inherent to Commiphora Myrrha (CM), Cobalt Ferrite Oxide (CFO), and Coal Fly Ash (CFA), as discerned through the analytical methodology of X-ray fluorescence (XRF). The outcomes of this analysis underscore that the triad of scrutinized samples predominantly exhibits iron (Fe) concentrations of 9.02%, 43.6%, and 0.893%, respectively. Furthermore, appreciable fluctuations in cobalt (Co) contents are evident, with percentages of 1.06%, 15.6%, and 0.0375% characterizing the respective constituents. Additionally, minor proportions of silica (Si) are identified, denoting 32.1%, 0.522%, and 2.82% in the designated order. Alongside these primary components, a range of diverse constituents such as sulfur (S), calcium (Ca), and nickel (Ni) are also present in varying quantities.

Table.1 Chemical Composition of CM, CFO, and CFA.

Compounds Formula	CM	CFO	Concentration
			(%) CFA
Si	32.1	0.522	2.82
Al	16.0	0.300	2.02
Fe	9.02	43.6	0.893
Ca	8.72	0.123	68.0
Co	1.06	15.6	0.0375
S	5.82	16.1	1.72
K	5.06	-	11.0
Cl	4.53	-	1.84
Ti	1.44	-	0.0877
Ni	1.89	0.102	0.136

3.2. Infrared Spectrum Results

3.2.1. Infrared spectrum of the Cobalt Ferrite Oxide (CFO)

The spectroscopic data acquired for the compound with the molecular composition of Cobalt Ferrite Oxide (CFO) reveals significant features in its infrared spectra. Evident within this spectral profile is a pronounced broad band manifesting at 3490 cm^{-1} , corresponding to the presence of water molecules. Furthermore, the identical spectrum exhibits a distinct band at 2600 cm^{-1} , indicative of the vibrational frequency (ν) attributed to the hydrogen-carbon double bond ($\nu\text{HC=O}$) inherent to the aldehyde moiety. In tandem, the infrared spectrum of the same compound evinces discernible bands positioned at 1480 cm^{-1} and 1625 cm^{-1} , which can be ascribed respectively to the characteristic vibrations of carbon-carbon double bonds ($\nu\text{C=C}$, aromatic) and carboxylic acid functional groups ($\nu\text{-COOH}$). The comprehensive infrared spectral presentation is visually depicted in Figure 2 for reference and analysis.

Table.2 Infrared spectral data of Cobalt Ferrite Oxide (CFO).

νOH	$\nu\text{C-H}$	$\nu\text{C=C}$	$\nu\text{HC=O}$	$\nu\text{-COOH}$	$\nu\text{Fe-O}$	$\nu\text{Co-O}$
3750 cm^{-1}	2950 cm^{-1}	1480 cm^{-1}	2600 cm^{-1}	1625 cm^{-1}	960 cm^{-1}	670 cm^{-1}

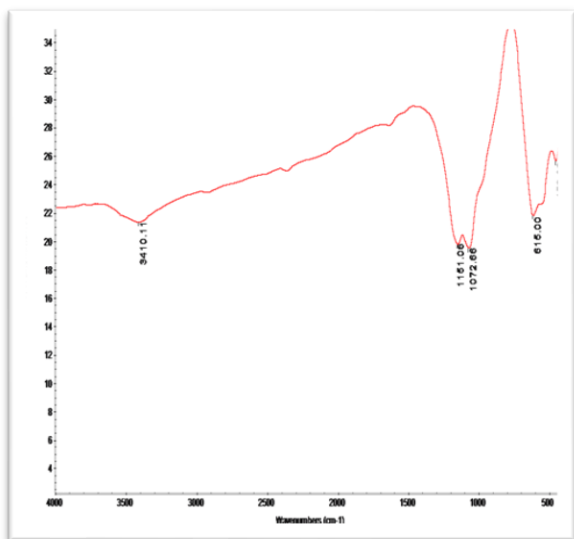


Figure .2. Infrared spectrum of the Nano Cobalt Ferrite Oxide (CFO).

3.2.2. Infrared spectrum of the *Commiphora Myrrha* (CM)

The spectral analysis conducted on *Commiphora Myrrha* unveils pertinent characteristics evident in its infrared spectra. A pronounced and broad absorption band is observed at 2966 cm⁻¹, which can be ascribed to the presence of water molecules. Similarly, within the same spectral context, a distinct band emerges at 1701 cm⁻¹, attributed to the vibrational frequency (ν) associated with the carbonyl functional group (ν C=O). Additional spectral features include a discernible band located at 1468 cm⁻¹, corresponding to the vibrational frequency of carbon-carbon double bonds (ν C=C) within the aromatic moiety. Concurrently, an observable band positioned at 1386 cm⁻¹ is attributed to the vibrational mode (ν) inherent to carboxylic acid functional groups (ν COOH). Further insights are garnered from the presence of a band at 1272 cm⁻¹ within the same spectrum, elucidating the occurrence of the hydroxyl group's (C-OH) vibrational mode.

Table.3 Infrared spectral data of *Commiphora Myrrha* (CM).

ν OH(H ₂ O)	ν C=O	ν C=C	ν COOH	ν C-OH
2966 cm ⁻¹	1701 cm ⁻¹	1468 cm ⁻¹	1386 cm ⁻¹	1272 cm ⁻¹

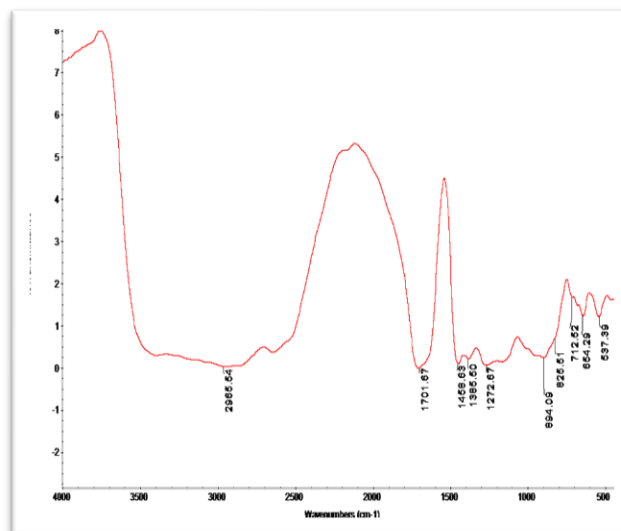


Figure 3. Infrared spectrum of the *Commiphora Myrrha* (CM).

3.2.3. Infrared spectrum of the Coal fly ash (CFA)

The infrared spectroscopic outcomes obtained from the analysis of Coal Fly Ash have been compiled and presented in Table 4. Within the recorded spectra of Coal Fly Ash, discernible features are apparent. A characteristic band at 3437 cm⁻¹ is observed, indicative of water molecules present in a hydrated state. Concurrently, these spectra manifest a prominent band at 2621 cm⁻¹, corresponding to the vibrational frequency (ν) attributed to the amino group (ν NH₂). Furthermore, a distinctive band is evident at 2967 cm⁻¹ within the same spectra, arising from the vibrational mode of aliphatic carbon-hydrogen bonds (ν C-H group). Similarly, the infrared spectrum of the aforementioned compound unveils an additional band located at 1446 cm⁻¹, which can be attributed to the vibrational frequency of carbon-carbon double bonds (ν C=C, aromatic). Moreover, the infrared spectrum depicts the presence of two distinct bands, as illustrated in Figure 4, signifying the vibrational modes of aromatic carbon-hydrogen bonds (ν C-H group, aromatic) and carbon-oxygen-hydrogen bonds (ν C-OH).

Table.4 Infrared spectral data of Coal fly ash (CFA).

ν OH(H ₂ O)	ν NH ₂	ν C=C	ν C-H	ν C-OH
3437 cm ⁻¹	2621 cm ⁻¹	1446 cm ⁻¹	2967 cm ⁻¹	1071 cm ⁻¹

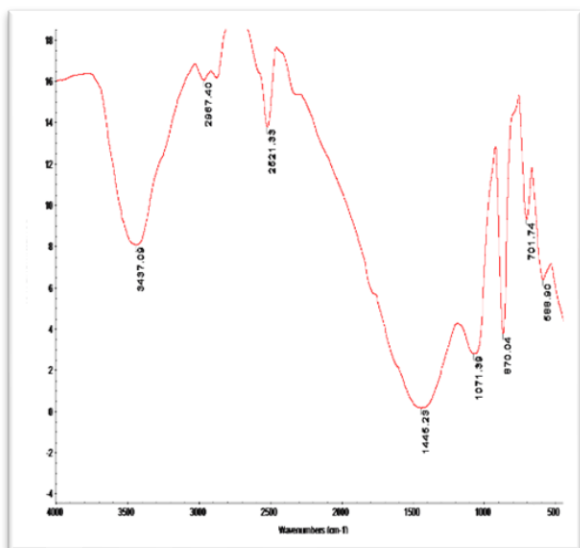


Figure 4. Infrared spectrum of the Coal fly ash (CFA).

3.3. XRD Analysis

The structural integrity and homogeneity in the phase of the CoFe_2O_4 synthesized using the sol-gel auto-combustion method were systematically verified through a comprehensive X-ray diffraction (XRD) analysis, as illustrated in Figure 5. The discernible diffraction peaks observed in the XRD pattern were meticulously cross-referenced with the established JCPDS (#22 1086) reference, thereby establishing their conformance with the face-centered cubic (FCC) crystalline configuration. The salient diffraction reflections correspondingly attributed to the (220), (311), (222), (400), (422), (511), and (440) crystallographic planes intrinsic to the CoFe_2O_4 spinel phase were unambiguously identified [32]. This coherent diffraction profile unequivocally confirms the singular presence of the cubic spinel structure, indicative of a pure and uniform phase formation within the synthesized specimens. Notably, the preeminent intensity peak, designated as (311), has been employed with specificity to deduce the characteristic size of the crystallites. The comprehensive X-ray diffraction analysis performed serves as a robust method for corroborating the successful synthesis of CoFe_2O_4 nanoparticles via the sol-gel auto-combustion process. The alignment of the observed diffraction pattern with the established reference further attests to the reliability and accuracy of the synthesized material's crystalline structure determination. Moreover, the distinct identification of diffraction reflections corresponding to specific crystallographic planes underscores the well-defined nature of the crystalline domains, affirming the consistent phase homogeneity throughout the synthesized samples. The characterization of crystallite size through the utilization of the most intense peak, i.e., (311), holds pivotal significance in providing insights into the nanostructured nature of the synthesized CoFe_2O_4 .

This approach enables the quantitative assessment of the dimensionality of the crystalline domains, contributing to a deeper understanding of the material's physical properties and behavior at the nanoscale. Consequently, the XRD analysis conducted not only validates the successful synthesis of CoFe_2O_4 but also furnishes critical structural and dimensional information, laying the foundation for subsequent investigations and applications in various scientific and technological domains.

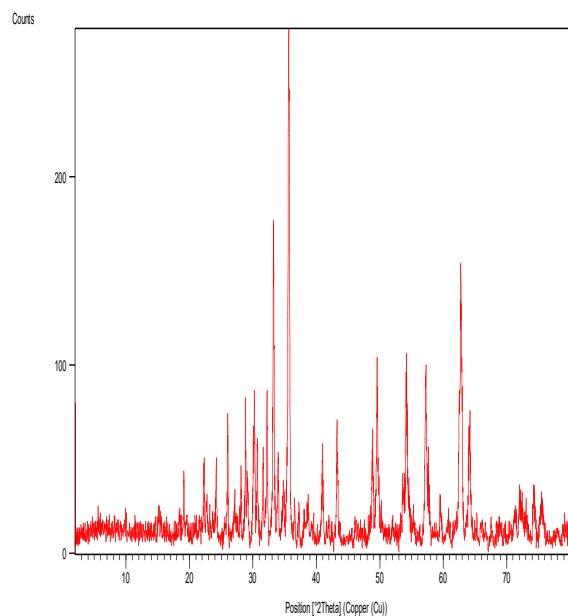


Figure 5. X-ray diffraction (XRD) pattern of the spinel cobalt ferrite oxide nanoparticles synthesized via sol-gel auto combustion method.

An insightful correlation between temperature variation and nanoparticle size augmentation is elucidated through the XRD analysis. This is exemplified by the observed tendency for particle size enlargement with increasing temperature, accompanied by concurrent mitigation of lattice imperfections and strains. This interplay is found to foster the coalescence of crystallites, thus leading to an elevation in the average nanoparticle dimensions [32]. Notably, at elevated temperatures, such as 600°C or beyond, the magnetization trends upwards in tandem with the temperature rise.

3.4. Seawater samples Results

Seawater samples were obtained from the coastal region of Libya, specifically Tripoli. The essential parameters characterizing the seawater were meticulously determined within the controlled environment of the Faculty of Science at the University

of Sebha. The quantified results of these parameters are comprehensively presented in Table 5.

Tab.5 Properties of seawater samples.

Type	Conductivity(mc/cm)	pH	TDS	Salinity (ppm)
Sea water	39.4	7.38	19311.89	21.615

3.5. Effect of Nano (CoFe₂O₄) with *Commiphora Myrrha* on crude oil removal %

Illustrated in Figure 6 is a conspicuous correlation delineating the interrelation between the degree of reduction in crude oil removal from seawater and the deliberate manipulation of particle size as applied to *Commiphora Myrrha*. The empirical findings presented in this visual representation unambiguously delineate a progressively enhanced effectiveness in the process of oil removal as a direct consequence of diminishing particle dimensions within the *Commiphora Myrrha* context. Notably, the Nano iteration of *Commiphora Myrrha* emerges as an exemplar of heightened efficiency in the realm of oil removal, particularly when considered within the framework of a specific composition ratio, denoted as 20:80. Within this compositional configuration, a notable spectrum of oil removal efficacy is conspicuously manifested, spanning from 22.63% to a substantial 30.70%. These delineations robustly underscore the exceptional proficiency of the Nano variant, firmly substantiating its preeminence in the crucial task of oil removal from seawater within the specified parameters of the investigation.

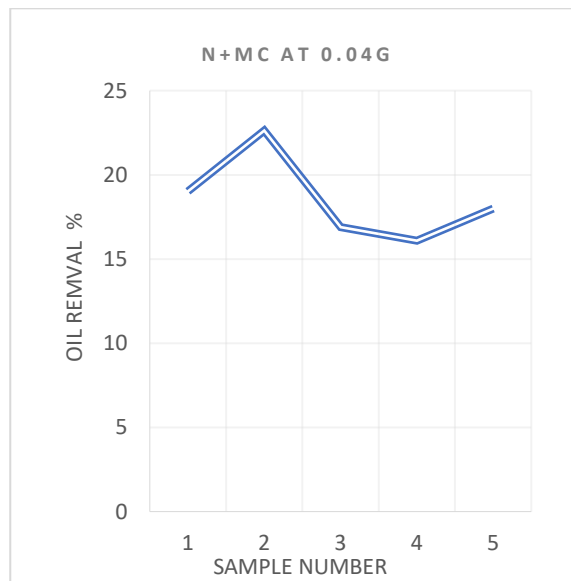


Figure 6. Removal efficiency percentage of Nano (CoFe₂O₄) with CM at 0.04.

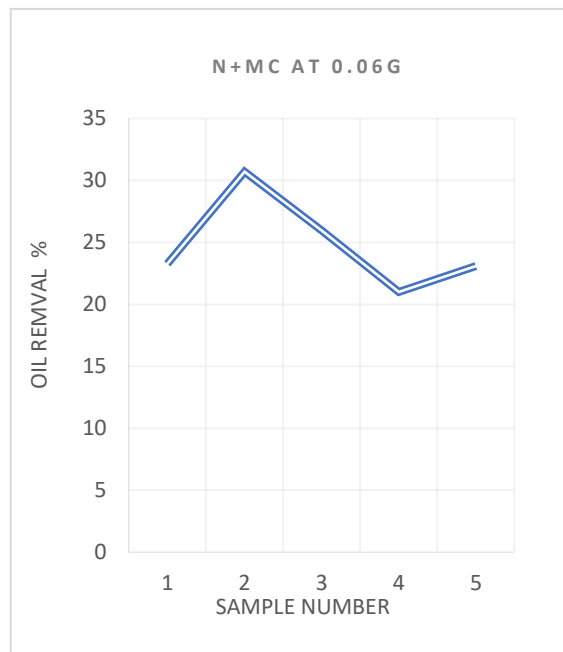


Figure 7. Removal efficiency percentage of Nano (CoFe₂O₄) with CM at 0.06.

3.6. Effect of Nano (CoFe₂O₄) with Coal fly ash on crude oil removal %

Illustrated Depicted in Figure 8 is a conspicuous and discernible pattern, wherein the proportional decrease in particle size attributed to coal fly ash correlates with an augmented reduction percentage concerning the elimination of crude oil from seawater. The empirical findings presented in this visual representation definitively delineate a progressive elevation in oil removal efficacy consequent to the utilization of nano coal fly ash. Remarkably, this heightened performance is most salient within the context of the 30:70 compositional ratio, where the recorded oil removal percentages span a range from 47.79% to a substantial 69.70%. The conspicuous augmentation in adsorption capacity observed when employing coal fly ash and its cognate counterpart, *Commiphora Myrrha*, can be attributed to the significant amplification in the ratio of exposed surface area to volume. This geometric alteration enables a more extensive interface for oil adsorption, thereby fostering an increased capacity for oil binding and coalescence on the material's surface. Consequently, this augmented adsorption capacity translates into heightened oil agglomeration and, in turn, superior efficiency in the removal of oil contaminants from the aqueous medium. The findings underscore the potential utility of tailored particle size manipulation in optimizing the performance of materials intended for oil removal applications. Such insights, rooted in materials

science and interfacial phenomena, not only advance the fundamental understanding of adsorption processes but also present practical implications for enhancing the efficacy of remediation strategies in the realm of marine oil pollution [33].

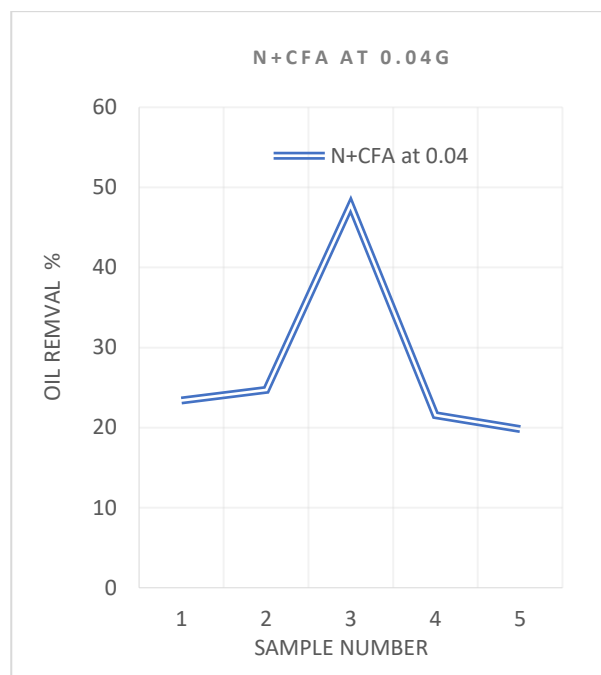


Figure 8. Removal efficiency percentage of Nano (CoFe₂O₄) with CFA at 0.04g.

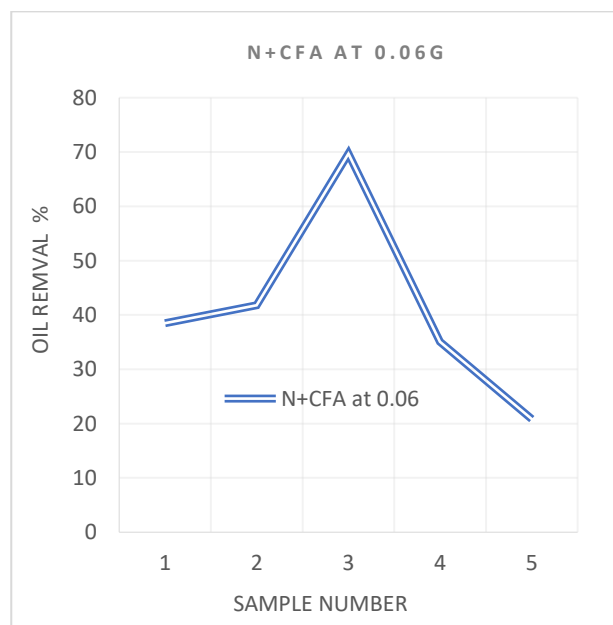


Figure 9. Removal efficiency percentage of Nano (CoFe₂O₄) with CFA at 0.06g.

4. Conclusion

The obtained results yield pertinent conclusions that merit elucidation. Firstly, it has been discerned that the utilization of Nanoparticles CoFe₂O₄ exhibits a notable efficacy in the removal of crude oil from seawater. Moreover, the strategic augmentation of Coal Fly Ash in conjunction with Nanoparticles CoFe₂O₄ has been identified as a viable approach to enhance absorbent capacity while concurrently mitigating cost implications. In a parallel vein, the integration of the natural substrate Commiphora Myrrha with Nanoparticles CoFe₂O₄ has yielded compelling outcomes in the domain of oil removal. Notably, this amalgamation has achieved substantial oil removal efficiency, quantified at an impressive 30.70%, particularly under the conditions of an 80:20 ratio at a concentration of 0.06g. Equally remarkable, the introduction of Coal Fly Ash as an absorbent agent has exhibited an even higher oil removal efficiency, registering an impressive 69.7%. This enhancement was most pronounced when adopting a 70:30 ratio at the same concentration of 0.06g. It is evident from these findings that the interplay between diverse materials and their ratios, along with concentrations, exerts a discernible influence on the overall efficacy of oil removal processes. Consequently, these findings not only contribute to advancing our understanding of effective oil spill mitigation strategies but also offer practical insights for optimizing the balance between performance and economic feasibility. Further research and explorations in this realm are encouraged to refine and expand upon these promising findings, thus fostering a more comprehensive framework for addressing oil contamination challenges in aquatic environments.

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