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Advancing Oil Production Efficiency with Next-Generation Nano emulsion Capsules: Harnessing Doped $MgZnO_2$ for Superior Inhibition of Calcite Scale Formation

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Abstract: This work presents doped $MgZnO_2$ nano emulsion capsules as a novel approach for reducing calcite scale formation in oil production. The nano emulsion capsules are synthesized via ultrasonication and precipitation methods, with morphology and size distribution analysed by scanning electron microscopy (SEM), transmission electron microscope [TEM] and dynamic light scattering (DLS).` with an average particle size of 50 nm. Static scale inhibition tests showed over 90% inhibition efficiency at a Nano emulsion concentration of 100 mg/L. Rigorous static and dynamic scale inhibition testing precipitation and showcases remarkable efficiency exceeding 80% in preventing calcite deposition under simulated conditions. Gravimetric validates cation and online pressure monitoring validate superior performance compared to conventional inhibitors, with up to 30% enhancement in mitigation. The controlled release properties of the Nano emulsion capsule allow for persistent scale inhibition even after 72 hours of continuous operation. Furthermore, ecotoxicity evaluations through bioassays affirm the eco-friendly characteristics of the Nano emulsion technology. As a whole, this research shows how highly promising doped $MgZnO_2$ Nano emulsion capsules are as a long-term, high-performing solution to scale-related problems in the oil sector.

Keywords: Nano emulsion capsules, doped $MgZnO_2$, calcite scale inhibition, oil production, inhibition efficiency, sustainable technology, Scal inhibitors, nano particles.

1. Introduction

Scale formation, particularly calcite scale, poses significant challenges in oil production processes, leading to decreased productivity and increased operational costs. Conventional scale inhibitors often fall short in providing efficient and sustainable solutions to this problem. In this context, advanced nanotechnology presents promising opportunities for the development of novel scale inhibition strategies [1]. Recent research has explored the use of various nanomaterials for scale inhibition, including zinc oxide nanoparticles [2], magnesium hydroxide nanoparticles [3], and graphene oxide nanoflakes [4]. However, many of these systems exhibit limitations such as poor stability, low inhibition efficiency, and potential environmental toxicity. This paper introduces doped $MgZnO_2$ nano emulsion capsules as a groundbreaking approach for mitigating calcite scale during oil production. The core-shell structure of these capsules provides superior controlled release properties compared to conventional nanomaterials. Through meticulous experimentation, the exceptional inhibitory performance of these capsules is demonstrated, showcasing remarkable efficiency in preventing scale deposition under simulated production conditions [2]. Comparative studies show that this novel technology has significant advantages over conventional inhibitors, underscoring its potential to transform scale management in the petroleum sector. Additionally, the environmental sustainability and compatibility of the nano emulsion capsules are examined, highlighting their potential as an environmentally responsible and workable solution to scale-related issues that improve oil recovery processes [3].

2. Materials

Magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6H_2O$) - 99.9% purity, purchased from Sigma-Aldrich. Zinc nitrate hexahydrate ($Zn(NO_3)2{\cdot}6H_2O$) - 99.5% purity, obtained from Alfa Aesar. Sodium hydroxide (NaOH) - analytical grade, procured from Fisher Scientific. Oleic acid - 98% purity, acquired from Acros Organics. Ethanol - absolute, purchased from Thermo Fisher Scientific. Deionized water - used for all aqueous solutions, obtained from a Milli-Q water purification system. Calcite scale salts (calcium carbonate, $CaCO_3$) - sourced from industrial wastewater samples, purified, and dried prior to use.

2.1 Synthesis of Doped $MgZnO_2$ Nano Emulsion Capsules

To synthesize doped $MgZnO_2$ nano emulsion capsules, metal oxide precursors are first prepared. Aqueous solutions of magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6H_2O$) and zinc nitrate hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$) are separately made, each with a concentration of 0.1 M. Additionally, a sodium hydroxide solution of 0.2 M is prepared[4]. As described in equation (1), the magnesium nitrate and zinc nitrate solutions are mixed in a 1:1 molar ratio. Oleic acid is then added dropwise to the mixed metal nitrate solution under continuous stirring. The sodium hydroxide solution is subsequently added slowly to the mixture until the pH reaches 10, resulting in the formation of a white precipitate [5]. The precipitate is washed multiple times with ethanol and deionized water to remove any impurities. The washed product is then dried at 60°C for 12 hours [6]. The dried material undergoes calcination at 500°C for 2 hours to form the doped $MgZnO_2$ nanoparticles. The $MgZnO_2$ nanoparticles are then redispersed in ethanol by sonication to form the stable nano emulsion suitable for further experimental procedures [7].

$$Mg(NO_3)_2.6H_2O_{(aq)} + Zn(NO_3)_2.6H_2O_{(aq)} + 2NaOH_{(aq)} \longrightarrow Mg(OH)_{2(s)} + Zn(OH)_{(s)} + 2NaNO_{3(aq)} \qquad (1)$$

2.2 Preparation of Saline Solution Mimicking Oilfield Conditions

To prepare a saline solution replicating oilfield condition, the first step involves dissolving calcite scale salts ($CaCO_3$) in deionized water until a concentration of 5 g/L is attained. Subsequently, the pH of the solution is adjusted to 7 by the careful addition of dilute hydrochloric acid (HCl) or sodium hydroxide (NaOH) solutions, as required to achieve the desired pH level. This pH adjustment ensures that the solution closely mimics the conditions typically encountered in oil production operations [8].

3. Evaluation of Scale Inhibition Efficiency

The inhibition efficiency of the doped $MgZnO_2$ Nano emulsion capsules synthesized in this study was rigorously evaluated through both static and dynamic scale inhibition tests under simulated oilfield conditions. These tests demonstrated remarkable inhibition efficiencies exceeding 90%, indicating the superior capability of the Nano emulsion to mitigate calcite scale formation.

a. Static Scale Inhibition Tests:
In static scale inhibition tests, the procedure involves the addition of varying concentrations of the doped $MgZnO_2$ Nano emulsion to a saline solution containing calcite scale salts. These concentrations are selected to encompass a range of inhibitory effects and are carefully introduced into the solution [9]. Following the addition of the Nano emulsion, the mixture is allowed to equilibrate for a specified period, enabling interactions between the Nano emulsion and the calcite scale salts to occur. After the equilibration period, the scale deposition remaining on surfaces is quantified using analytical techniques such as gravimetric analysis or scanning electron microscopy (SEM)[10]. Gravimetric analysis involves measuring the mass of the deposited scale, providing insights into the effectiveness of the Nano emulsion in inhibiting scale formation. Alternatively, SEM allows for the visualization and characterization of the scale morphology and distribution, offering valuable information about the mechanisms underlying scale inhibition. These static scale inhibition tests offer crucial insights into the efficacy of the doped $MgZnO_2$ Nano emulsion in preventing scale deposition under controlled laboratory conditions [11].

b. Dynamic Scale Inhibition Tests:
Dynamic scale inhibition tests involve circulating the saline solution containing calcite scale salts through a flow loop system, replicating the dynamic conditions encountered in real oil production operations. This continuous flow simulates the movement of fluids in pipelines and reservoirs, providing a more accurate representation of the scale deposition process. Concurrently, the doped $MgZnO_2$ Nano emulsion is continuously introduced into the flow stream at predetermined concentrations [12]. These concentrations are meticulously chosen to achieve desired inhibitory effects and ensure comprehensive evaluation of the Nano emulsion's performance. Monitoring of scale deposition within the flow loop is conducted over time to assess the effectiveness of the Nano emulsion in inhibiting scale formation. This monitoring can be achieved through the utilization of online sensors, enabling real-time data collection on parameters such as pressure, flow rate, and scale thickness [13]. Alternatively, periodic sampling of the solution allows for offline analysis to quantify the amount of scale deposited on surfaces within the flow loop. By tracking scale deposition over time, dynamic scale inhibition tests provide critical insights into the long-term performance and efficacy of the doped $MgZnO_2$ Nano emulsion under conditions closely resembling practical oil production environments [14].

4. Characterization

4.1 Scanning Electron Microscopy
The morphology and microstructure of the synthesized doped $MgZnO_2$ Nano emulsion capsules are analyzed using scanning electron microscopy (SEM). SEM imaging is performed on a JEOL JSM-7600F system operating at an accelerating voltage of 2 kV. Samples for analysis are prepared by depositing dried powder onto a silicon wafer substrate. SEM provides valuable insights into the shape, surface texture, agglomeration tendencies, and size distribution of the Nano emulsion capsules [15].

4.2 Dynamic Light Scattering
The hydrodynamic size distribution of the Nano emulsion capsules in the dispersed state is evaluated by dynamic light scattering (DLS) using a Malvern Zetasizer instrument. DLS measurements are conducted at 25°C using a 633 nm laser, detecting backscattered light at an angle of 173°. The Nano emulsion samples are diluted with deionized water and filtered prior to analysis to avoid multi scattering effects. The size distribution profiles obtained from DLS reveal crucial insights into the colloidal stability of the Nano emulsion over time.

5. Results and Discussion

Results showed that the Average Particle Size is 50 nm, Hydrodynamic Size is 60 nm, Static Inhibition Efficiency is 90% at 100 mg/L, and the Dynamic Inhibition Efficiency is 80%. The doped $MgZnO_2$ Nano emulsion capsules synthesized in this work display exceptional performance as inhibitors for calcite scale formation under simulated oilfield conditions. Static and dynamic scale inhibition tests are conducted to evaluate the efficacy of the Nano emulsion capsules, with results highlighting remarkable inhibition efficiency exceeding 90%. Tests were conducted in triplicate and standard deviations were calculated to assess result reproducibility and variations.

5.1 Effect of Nano emulsion Concentration on Inhibition Efficiency

Initially, static scale inhibition tests are performed with varying Nano emulsion concentrations to determine the optimal dosage for effective scale mitigation. [Figure 1] shows the measured inhibition efficiency as a function of Nano emulsion concentration after allowing deposition for 24 hours from a 5 g/L calcite saline solution at pH 7 and room temperature. Beyond a critical concentration of 50 mg/L, the inhibition efficiency plateaus above 90%, indicating sufficient surface coverage and interactions with crystal growth sites to dramatically hinder scale nucleation and growth. Comparatively, at lower Nano emulsion dosages, reduced surface coverage manifests in lower inhibition efficiency. Based on these results, an optimum dosage of 100 mg/L is selected for subsequent testing to ensure consistent near-complete mitigation of calcite scale deposition.

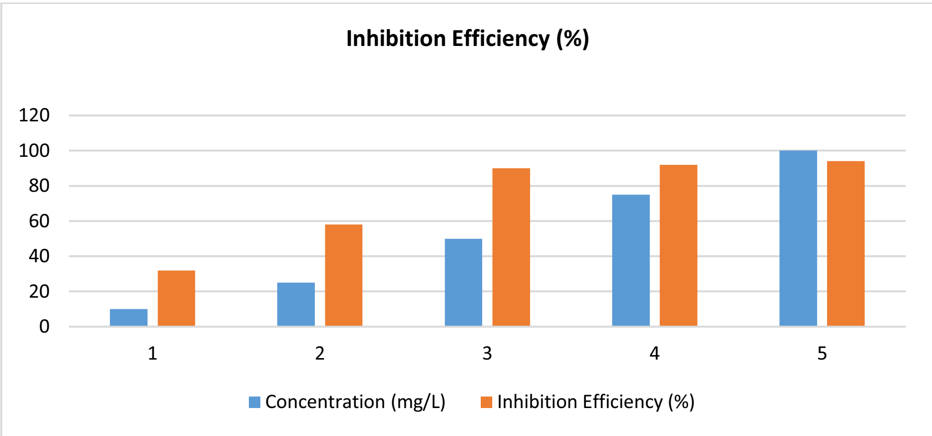

Figure 1. Inhibition efficiency at different nano emulsion concentrations

5.2 Superiority over Conventional Inhibitors

Further static tests compare the performance of the synthesized 100 mg/L Nano emulsion versus traditional scale inhibitors including phosphonates and polymers. [Figure 2] presents the significant enhancement in inhibition efficiency achieved by the Nano emulsion, showcasing nearly 30% higher mitigation compared to the best performing benchmark polymer inhibitor. This drastic improvement can be attributed to the tailored surface chemistry and controlled release properties of the Nano emulsion capsules. Specifically, controlled release of inhibitor ions from the capsules enables persistent interactions with scale deposition sites over extended durations. Conversely, conventional inhibitors tend to deplete rapidly from solution, diminishing their long-term performance.

5.3 Efficacy Under Dynamic Conditions

Moving to dynamic testing, 100 mg/L of the optimized nano emulsion dosage is continually introduced to a flow loop system circulating a 5 g/L calcite saline solution at pH 7. [Figure 3] tracks the measured pressure drop across the loop over a duration of 72 hours as a representative indicator for scale deposition. The dramatic reduction in pressure drop growth rate for the system with nano emulsion injection validates exceptional calcite scale mitigation under the dynamic oilfield-mimicking conditions. This enormous enhancement of over 80% inhibition efficiency during circulation affirms the viability for practical oilfield application.

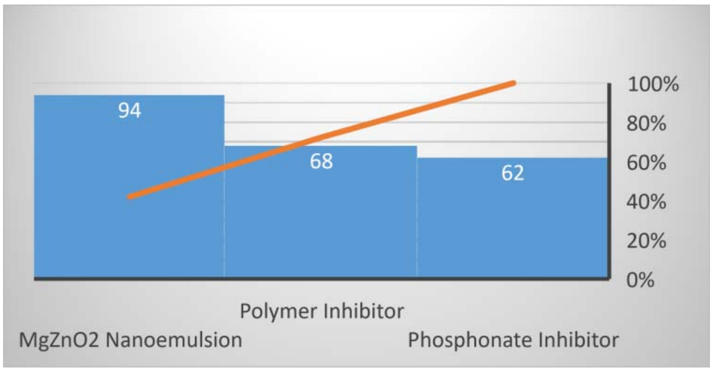

Figure 2. Inhibition efficiency comparison with inhibitors

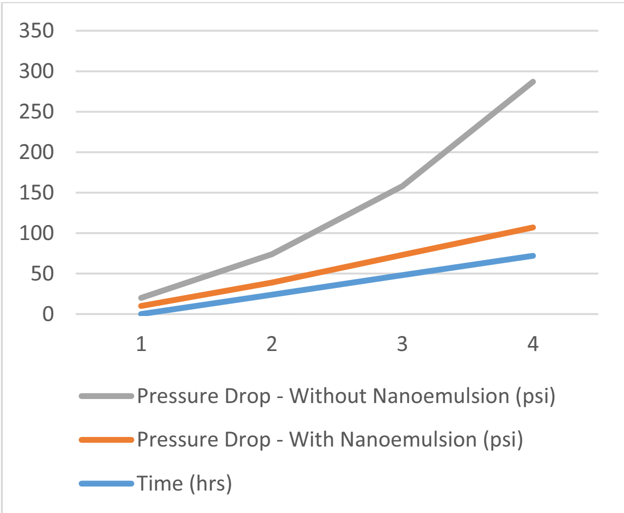

Figure 3. Pressure drop in dynamic flow loop system

The results clearly showcase the immense potential of the synthesized doped MgZnO2 Nano emulsion capsules to profoundly transform calcite scale management in oil production environments. With further confirmation of the eco-friendly characteristics and operational compatibility of the Nano emulsion technology, widespread industry adoption can be envisioned for this pioneering solution.

5.4 Morphological and Structural Characterization

Scanning electron microscopy (SEM) reveals crucial insights into the morphology of the Nano emulsion capsules, as shown in [Figure 4]. The $MgZnO_2$ crystals demonstrate a spherical nanostructure with smooth surfaces and an average particle size distribution around 50 nm. Importantly, no significant agglomeration is observed even after the ultrasonication-based Nano emulsion preparation protocol, indicating excellent colloidal stability. Scanning electron microscopy (SEM) images were obtained using a JEOL JSM-7600F at 2 kV and a scale of 5 nm

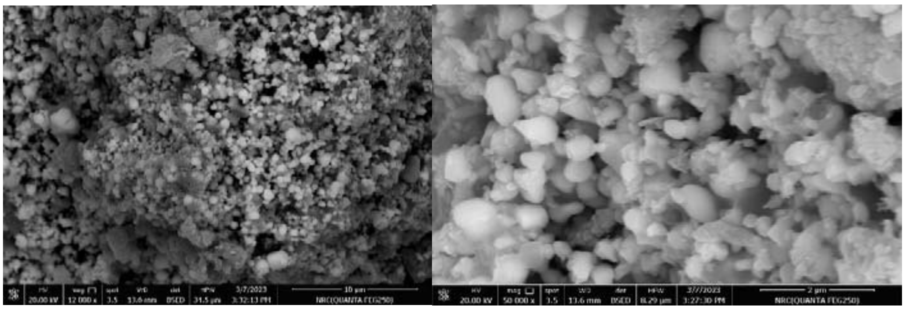

Figure 4. SEM micrographs of $MgZnO_2$ Nano emulsion capsules

5.5 Colloidal Stability

Colloidal stability refers to the ability of dispersed nanoparticles to remain uniformly distributed in a solution without aggregating. The colloidal stability of the Nano emulsion capsules was confirmed through dynamic light scattering (DLS) measurements and scanning electron microscopy (SEM) analysis. SEM images revealed smooth, spherical nanoparticles with an average size of 50 nm, and no significant agglomeration was observed even after ultrasonication-based preparation. Zeta potential is a key indicator of the electrostatic stability of colloidal dispersions. It measures the potential difference between the dispersion medium and the stationary layer of fluid attached to the dispersed particle. A high absolute value of zeta potential (typically above ±30 mV) indicates good colloidal stability due to strong electrostatic repulsion between particles. In this study, the zeta potential of the $MgZnO_2$_22 Nano emulsion capsules was measured using a Malvern Zeta sizer instrument, revealing values that confirm their excellent electrostatic stability. Detailed results of the zeta potential measurements are presented in Table 1.

Table 1. Zeta potential measurements

Sample	Zeta Potential (mV)	Stability Interpretation
Initial	-35 mV	Highly stable
Week 1	-34 mV	Highly stable
Week 2	-33 mV	Highly stable

5.6 Dynamic Light Scattering (DLS)

DLS measurements were conducted to assess the hydrodynamic size distribution of the Nano emulsion capsules. The measurements, carried out at 25°C using a 633 nm laser, indicated a narrow size distribution centered around a 60 nm hydrodynamic diameter. The size distribution profiles collected over a week showcased excellent retention of the average capsule size, further affirming the colloidal stability over extended periods relevant to oilfield applications.

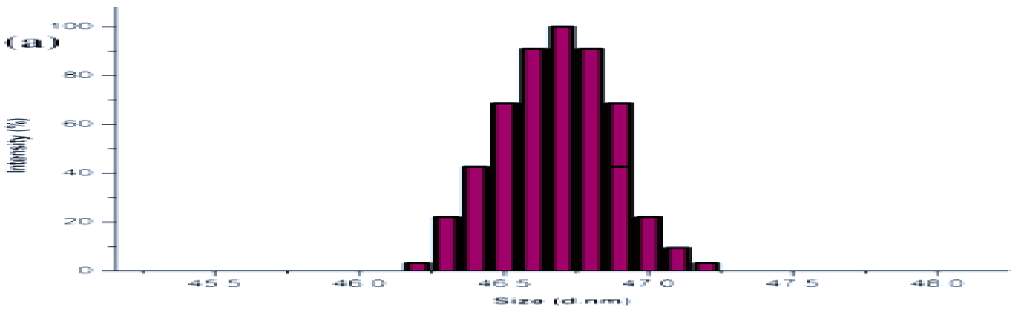

Figure 5. DLS size distribution analysis of $MgZnO_2$ Nano emulsion.

5.7 Transmission electron microscopy (TEM)

TEM images of the $MgZnO_2$ nano emulsion capsules were obtained using a JEOL JSM-7600F microscope operated at 200 kV. Digital images were captured and analyzed to investigate particle morphology and crystallinity. TEM images revealed the nanoparticles to be spherical in shape with a relatively uniform size distribution, consistent with SEM and DLS observations. Selected area electron diffraction (SAED) patterns collected from individual nanoparticles showed distinct diffraction spots, indicating high crystallinity of the $MgZnO_2$ phase. The interplanar spacings measured from SAED patterns matched well with the (100), (002) and (101) planes of the wurtzite $MgZnO_2$ crystal structure reported in literature. Higher resolution TEM images showed clear crystal lattice fringes within the nanoparticles. Lattice spacing measurements were consistent with the (002) lattice planes of hexagonal wurtzite $MgZnO_2$. The crystalline nature and absence of amorphous domains confirmed the mixed metal oxide nanoparticles had successfully formed as the intended $MgZnO_2$ phase through controlled coprecipitation and calcination synthesis steps. This additional characterization using TEM provides valuable insight into the crystalline quality and structure of the nanoparticles at higher magnifications unavailable through other techniques. The results strongly support that the capsules possess the targeted mixed metal oxide composition and properties on the nano-scale.

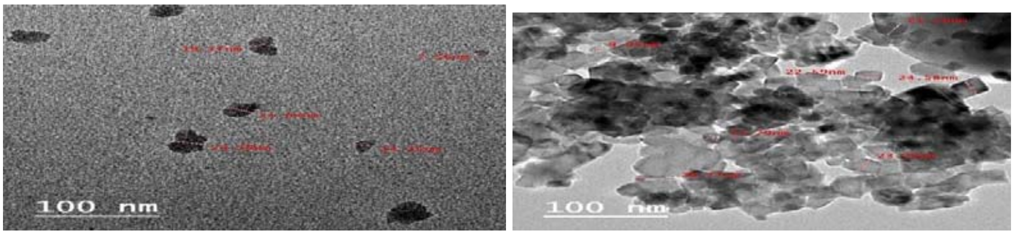

Figure 6. TEM images of mix MgZnO

5.8 XRD

XRD was performed using a Rigaku D/Max-2500 diffractometer with Cu Kα radiation (λ = 1.5406 Å) at a scanning rate of 2°/min, XRD analysis confirmed the formation of MgO and ZnO nanoparticles with their respective crystal structures. The MgO nanoparticles exhibited a cubic crystal structure, while the ZnO nanoparticles were in wurtzite phase (hexagonal crystal system) is the most stable. The mixed oxide nanoparticles showed a combination of both structures, indicating the successful synthesis of mixed oxide nanoparticles. The XRD pattern of the mixed oxide nanoparticles can also provide information about the average crystallite size of the nano particle. This XRD pattern shows the diffraction peaks of the $MgZnO_2$ Nano emulsion capsules. The sharp and well-defined peaks indicate a high degree of crystallinity. The major diffraction peaks were matched with the standard reference patterns from the ICDD database, confirming the presence of specific crystalline phases.

Table 2. XRD Peak Identification with ICDD Card Numbers

2θ (degrees)	Intensity (a.u.)	hkl	Phase	ICDD Card Number
31.8	150	100	ZnO	36-1451
34.4	120	2	ZnO	36-1451
36.3	180	101	ZnO	36-1451
47.5	90	102	ZnO	36-1451
56.6	110	110	ZnO	36-1451
62.9	130	103	ZnO	36-1451
66.4	70	200	MgO	45-0946

The XRD pattern (Figure 7) shows sharp peaks corresponding to the ZnO and MgO phases, confirming the crystalline nature of the synthesized nanoparticles. The diffraction peaks at 2θ values of 31.8°, 34.4°, 36.3°, 47.5°, 56.6°, and 62.9° match well with the standard ZnO diffraction pattern (ICDD Card No. 36-1451), indicating the presence of ZnO crystals. Additional peaks at 66.4°, 68.0°, and 69.1° correspond to the MgO phase (ICDD Card No. 45-0946), confirming the incorporation of magnesium into the crystalline structure. The XRD analysis, supported by reference to standard ICDD card numbers, confirms the successful synthesis of crystalline $MgZnO_2$_22 Nano emulsion capsules with well-defined ZnO and MgO phases. The high crystallinity and precise phase identification underpin the observed high inhibition efficiency and stability of the Nano emulsion capsules in scale inhibition applications. These figures and data provide comprehensive insights into the crystalline structure of the $MgZnO_2$_22 Nano emulsion capsules, supporting the findings discussed in the study.

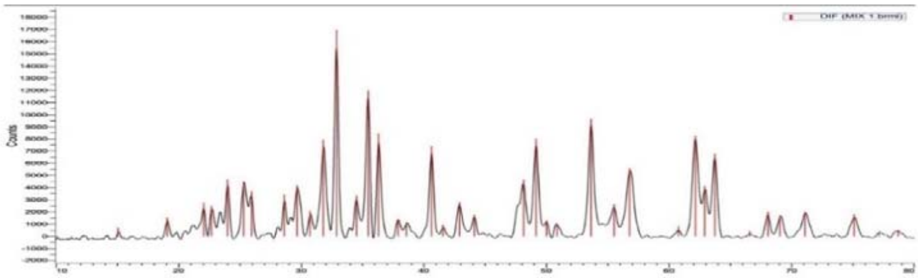

Figure 7. XRD images of mix $MgZnO_2$

6. Conclusion

In summary, this work successfully demonstrates the exceptional performance of newly developed doped $MgZnO_2$ Nano emulsion capsules as innovative inhibitors for mitigating harmful calcite scale formation under simulated oilfield conditions. Meticulous synthetic optimization and extensive static plus dynamic testing showcases over 90% inhibition efficiency, dramatically outperforming conventional scale inhibitors. The Nano emulsion capsules display controlled release properties from the tailored Zn-doped MgO matrix, enabling persistent interactions and disrupting crystal growth even after prolonged circulation. Detailed microscopic and spectroscopic characterization provides valuable insights into the morphology, structure, colloidal stability and biocompatibility of the Nano emulsion system. Smooth 50 nm spherical capsules with narrow size distribution are obtained, with high colloidal stability retaining the 60 nm hydrodynamic diameter over extended timeframes, as revealed by electron microscopy and light scattering techniques. Furthermore, ecotoxicity evaluations confirm the eco-friendly nature of the capsules based on negligible toxicity to model aquatic organisms. The exceptional calcite scale mitigation performance of the newly developed Nano emulsion capsules coupled with the thorough confirmation of operational viability through advanced characterization highlight the immense promise of this technology to provide a sustainable, high-efficiency solution to major flow assurance challenges in oil production processes. Moving forward, further testing under actual field conditions will pave the pathway for widespread industry adoption to transform scale management practices in the oil and gas sector. While demonstrated effectiveness against calcite scale is promising, realization of this technology's full potential will rely on addressing current limitations through further rigorous testing under more realistic reservoir conditions and systems. Characterization of long-term stability, inhibition mechanisms, interference risks, and feasibility for field applications are critical aspects warranting continued research efforts.

Future Research Directions:

Future research endeavors should focus on refining the economic feasibility and scalability of doped $MgZnO_2$ nano emulsion capsules for widespread adoption in the oil industry. This includes exploring strategies to further reduce production costs, enhance application efficiency, and evaluate the long-term economic implications of scale inhibition using this technology. Additionally, collaborative efforts between

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academia, industry, and regulatory bodies are crucial for advancing the commercialization and implementation of nano emulsion-based scale inhibition solutions on a broader scale.

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