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Feasible calcium carbonate polymorphs synthesis from natural corallines and their potential gelling role in hydraulic fracturing application

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ABSTRACT

Hydraulic fracturing fluids are significantly important for petroleum and related industrial areas in the preceding and forth-coming eras. In this work, we have feasibly synthesized the calcium carbonate polymorphs from the natural coralline limestones through a chemical precipitation process. The apparent physical morphology of the structure revealed the co-existence of the aragonite, vaterite, and calcite within the structure while the aragonite is the major component throughout the structure. The X-ray diffraction and Zeta sizer showed a nominal particle size of 23 nm of the structure which is well-matched with the prior reports. The acquired polymorphs have been functioned for the preparation of the gelling agent. The physicochemical characteristics of the agent were implemented in the materials safety data sheet of the product. The Gelling agent in the report is compared to familiar agents based on Xantham gum which reflects much less cost as well as the proposed potential application in the medical and petroleum industry.

1. INTRODUCTION

Science is advancing progressively in the few preceding decades to establish solutions for industrial and health-related applications. These applications aim to enhance the everyday activity of people, raise national income, and limit the side effects of pollution and accompanying environmental changes [1]. The population of humans all over the world is continuously increasing which in turn reflects environmental challenges such as global warming, environmental contamination, and non-renewable energy sources consumption [2]. The integration of naturally existing abundant materials in the industrial sector satisfies the objective of the highly increasing demand for these solutions. Calcium carbonates are abundant white insoluble powdery material existing naturally in chalk, minerals, marbles, limestones, shells, and else.

Calcium carbonates are characterized by their abundance which comprises three crystalline polymorphs. (i.e., aragonite, calcite, and vaterite) [3,4] The thermodynamics viewpoint reveals the highest stability of the rhombohedral calcite phase and the reduced stability of the hexagonal vaterite phase [5]. The calcium carbonates might be presented to production using numerous methods and chemical precursors such as the sonochemical approach [6], co-precipitation [7], and microwave-aided procedure [8]. Calcium carbonates deliver numerous potential applications in fillers, paints, plastics, cosmetics, water treatment, drug delivery systems, the food industry, and so on [9–14] These potential applications are promoted by the fact of the enriched and elevated surface area, dispersion, biocompatibility, biodegradability, and porosity. Coralline limestones are biological grainy rocks, composed basically of calcium carbonate, their specific composition might vary depending on the origin and the neighboring location [2]. These coralline limestones have resided from ancient ages in plentiful amounts with scarcely limited employment in desirable industrial areas despite their rich calcium carbonate content [15]. One of the major industrial challenges in the petroleum sector is the hydraulic fracturing application [16].

Hydraulic fracturing combined with horizontal drilling permits the industrial sector to access the entrapped and inaccessible reservoirs of gas and oil energy supplied in geologic formations. This process demands enlarged amounts of high-pressure water and fluids which need to be safely and appropriately delivered to the application target.

Hydraulic fracturing fluid's first experiment involved gasoline gelation with palm oil and naphthenic acid during the Second World War and was referred to as Nalpalm [16].

Since that time, numerous efforts have been conducted to deliver hydraulic fracturing gels that satisfy a desirable set of requirements to limit sand production and more. These requirements include -but are not limited to biosafety, non-damaging fracture conductivity or reservoir permeability, versatility, and cost-effective materials. Fluids with water as their primary component are desired. Most gels used in fracturing are composed of polymers such as guar gum and Xantham gum. These polymer-based gels have shortcomings such as high residual after breaking, and limited resistance to temperature variation [17]. In fracturing, fluids are pumped down to create fractures and to provide accessible transportation of proppant sand to maintain the open state of the fracture. In general, these gelling agents are important for industry and more specifically for the petroleum industry. Petroleum companies incorporate huge amounts of gelling agents as a carrier for sand and fluids delivered to oil fields (hydraulic fracturing). It consumes 40-100 k gallons of the gelling agent through the job performed for 4-5 days. The incorporation of nanoparticles into polymer or gel-based fracturing fluids might improve the internal network structure of the fluid and its accompanying specifications such as rheology, temperature uptake, and stability [17].

The ideal gelling agent should satisfy at most all or a few of these requirements: non-toxic, viscous, cost-effective, biosafe, and eco-friendly, easy to prepare, non-reacting with the surrounding medium, Stable at high temperatures, have a basic nature, doesn’t cause agglomeration, doesn’t need the addition of oxidative agent such as ammonium persulphate to eradicate the free radicals. Few preceding efforts are delivered for the formation of gelling agents, Leiming Li et al., synthesized the aluminum pillared montmorillonite clay additive and incorporated them in oil fluid gels which reflect enhanced viscosity and stability at raised temperatures [18]. Additionally, R. V. V. Ramana Murthy, et al., successfully prepared a novel designed linear gel for unconventional wells [19]. In another research, Rajendra A. Kalgaonkar, et al., developed a nanoparticle-based- gel that suppresses the inherent challenges of conventional crosslinked based compositions and sustains employment in 20% HCl and around 100 ºC providing less prone damage formation in comparison with a crosslinked polymer-based compositions [20]. Muhammad Shahzad Kamal, et al., acquired a smart fracturing gel that is tunable for low or high pH values [21].

Despite these efforts and as far as the authors are aware, no efforts were conducted to functionalize the coralline limestones with their intrinsic properties in the synthesis for the cost-effective and rapid formation of the gelling agent.

In this work, instead of the existence of these abundant corralines without employment. We feasibly synthesized the calcium carbonates from naturally abundant coralline limestones on the Red Sea Coast. We employed these carbonates in the synthesis of eco-friendly, rapid formation of a gelling agent that is beneficial and applied in the hydraulic fracturing process. The structural and morphological investigation of the prepared gelling agent was inspected. To satisfy the growing demand of uncommon solutions, this gelling agent might provide insight into the petroleum industry as well as industrial applications.

2.MATERIALS AND METHODS

2.1 Materials

Acetic acid, (Sigma Aldrich, CH_3CO_2H, M.wt: 60.05, 99.7%), naturally existing coralline limestone (porites), sodium hypochlorite (Sigma Aldrich, NaClO M.wt: 74.44,10%), and triethanolamine (Sigma Aldrich, $(HOCH_2CH_2)_3N$, M.wt: 149.19, 98%), Poly(ethylene glycol, PEG 400, Sigma Aldrich), $(H(OCH_2CH_2)_nOH)$, M.wt 400).

2.2 Preparation of polymorphs from coralline material (porities)

Old limestones (Pleistocene age) from the Red Sea Coast in Egypt were acquired as it is and employed for the further steps of preparation. Limestone fragments were cleaned with a brush to remove dust. Then it was immersed in sodium hypochlorite solution (0.2 g/ 100 mmol water) for 12 h for surface purification. Sodium hypochlorite is commonly used as a disinfecting agent for the eradication of dirt, stains, and other contaminations. Finally**,** the limestone fragments were rinsed with flowing water and dried in an open space to complete the drying. The cleaned pieces were added into a Pyrex container and partially soaked in acetic acid solution (10%) for 10 days at room temperature and atmospheric pressure, which is a satisfactory duration to complete crystals growth of polymorphs on the entire priorities surface. The growth of the polymorphs is enhanced by the exposure of the surface to sunlight (5 h/day) which provides activation energy. This agrees with previously reported research [22–26] Like so, the propagation of rod-shaped calcium carbonates on the surface progressed with decent producibility and without further impurities or contamination. This growth initiates after 4 days of immersion. In addition to the collected polymorphs from the surface, the residuals in the acetic acid solution were also collected and dried in an oven (180 ºC, 2 h) to eliminate the solution and maintain the dried calcium carbonates. Both calcium carbonates were gathered and grounded using a lab mortar and pestle to a fine powder and sieved using 212 µm (70 mesh) sieves which were ready for further physical investigation. Fig.1 represents the scheme for the polymorph's preparation.

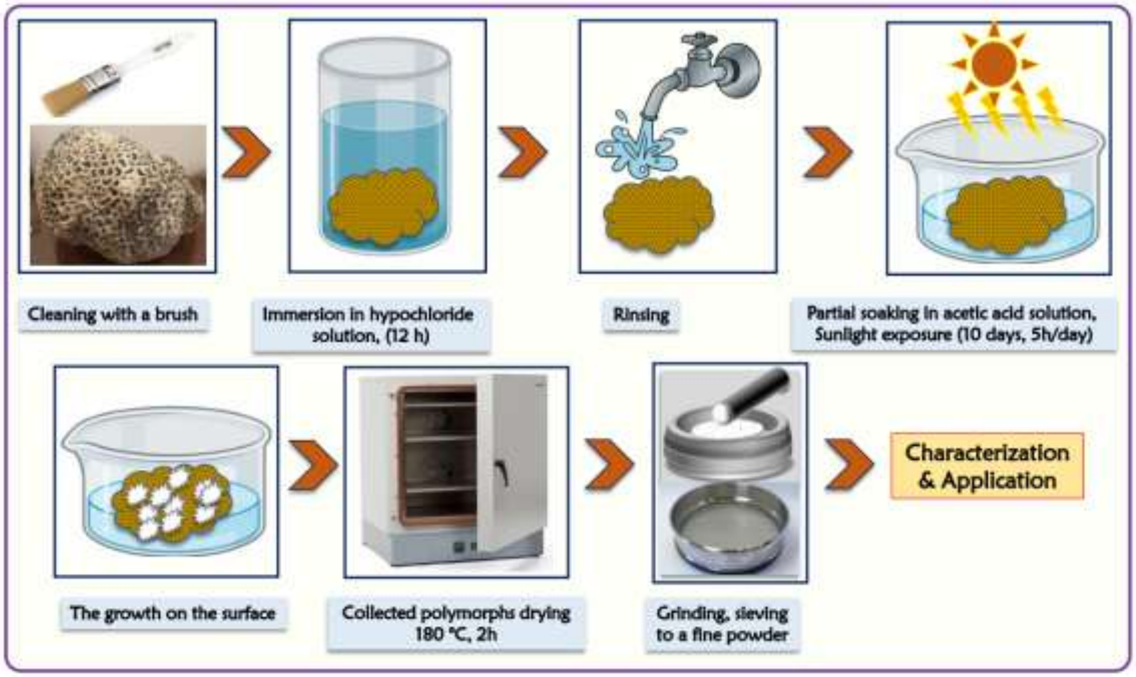


Figure 1. Procedures used for the preparation of the Polymorphs. Starting with the coralline limestone, heading to the meersion, and finally seizing a powder ready for further analysis.

2.3 Synthesis of gelling agent based on calcium carbonate polymorphs

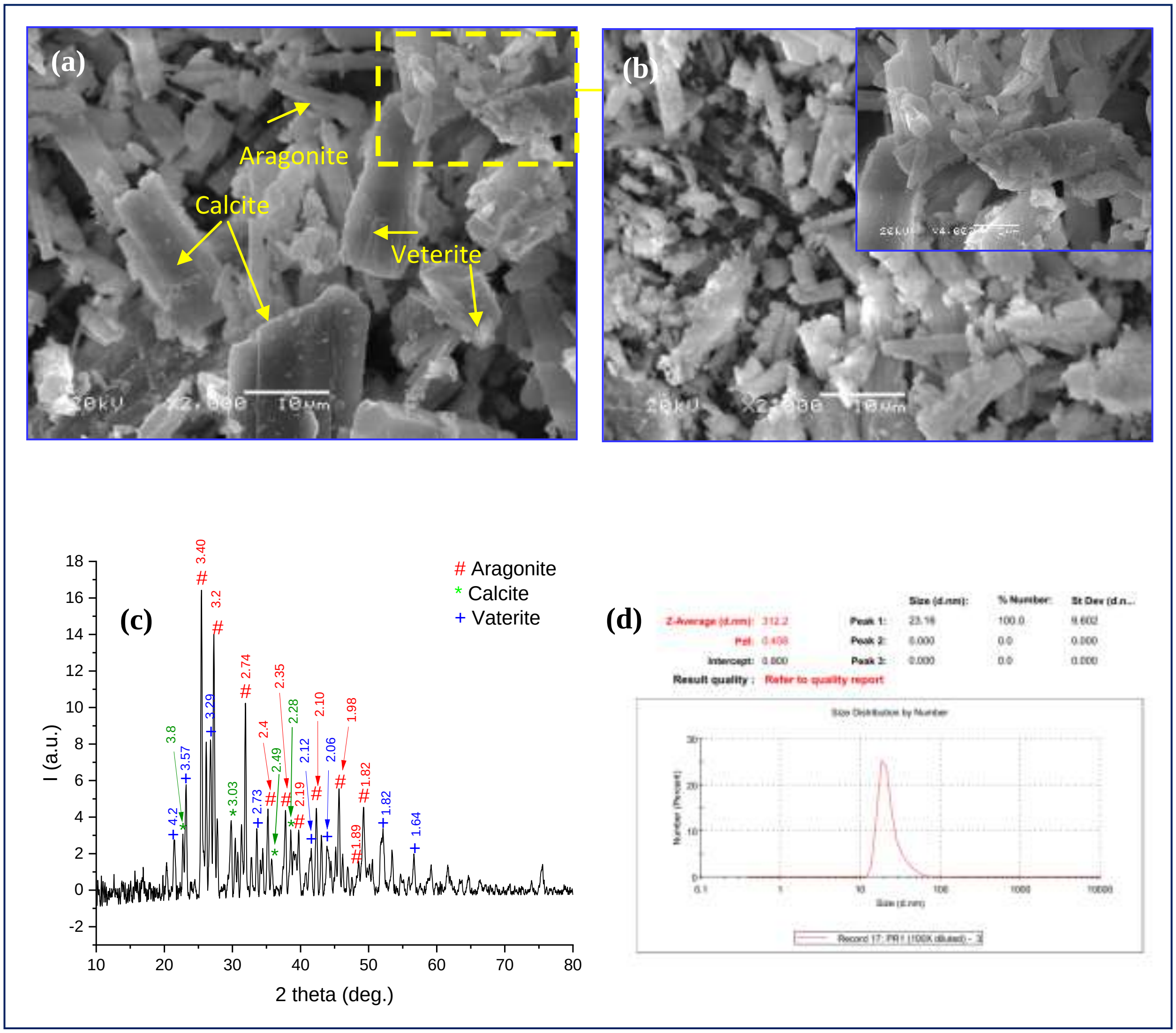
Next to the acquisition of the calcium carbonate-based polymorphs from coralline limestones **(**porities**),** the gelling agent was feasibly prepared as follows: 2.5 g of calcium carbonate polymorphs were mixed in a glass beaker with 8.5 ml of deionized water and stirred for 15 minutes on a magnetic stirrer (without heating) to enhance the distribution of the particles in the medium and obtain a slurry, following to this step, 60 ml of triethanolamine as a drop-wise addition under sustained stirring which promotes the formation of the gel after few seconds and the addition continues until the completion of the 60 ml drop-wise insertion for around 2-3 min and then the entire solution transforms into the gel state. The triethanoleamine is reported to maintain viscosity and stability and works as a fluid stabilizer [27]. Another gel was prepared by an identical approach replacing only 60 ml of triethanolamine with 16 ml of polyethylene glycol solution. The PEG enhances the apparent viscosity and functions as a solvent, besides it works as an anti-sedimentation agent [28]. These materials were used as it is related to industrial application areas as surfactant material as well as their environmentally safe nature [29]. The selection of the appropriate weights relied on a set of trials and previously reported research for other materials [30].

2.4 Characterization

X-ray diffraction (XRD) Rigaku with a CuKα incident light source (1,54 Å wavelength) was exploited to reveal the structural specifications of the synthesized polymorphs with a scanning rate of 2º per minute from $2\theta=10$ º up to 80º. A scanning electron microscope (SEM, JEOL 7600F) was used to depict the polymorph’s apparent morphology operated at 20 kV. Particle size distribution was also assessed by Zeta sizer (Malvern, UK) through Dynamic light distribution (DLS).

3.RESULTS AND DISCUSSION

3.1 Physical characterization



Z-Average (d.nm): 312.2
PdI: 0.408
Intercept: 0.800
Result quality : Refer to quality report

	Size (d.nm):	% Number:	St Dev (d.n...
Peak 1:	23.16	100.0	9.602
Peak 2:	0.000	0.0	0.000
Peak 3:	0.000	0.0	0.000

Figure 2. (a), (b) the Scanning electron microscope (SEM) micrographs of the synthesized polymorphs, (c) the X-ray diffraction of the prepared polymorphs (d-spacing values are introduced over each peak), and (d) the particle size distribution of the acquired polymorphs developed by Zeta sizer.

The apparent physical specifications of the synthesized calcium carbonates are represented in Fig. 2. The conventional morphology of the prepared carbonates is elucidated in (Fig. 2a), and (Fig. 2b), at different magnifications. The micrograph morphology reflects the co-existence of the rod-like shape of the aragonite as well as the rhombic-shaped calcite crystals along with the distributed round vaterite amongst the structure. The possible irregularity of the formed sizes and morphologies might be pursued by the differences in preparation circumstances [31,32] or additional surfactants [33–35]. As reported previously, the surfactants either ionic or nonionic can change the size and morphology of the emerged calcium carbonates [4,36]. The acquired morphological outcomes are reported earlier and our results resemble the previous research which validates the accuracy of the preparation approach [37,38]. The three formed polymorphs (i.e. calcite, aragonite, and vaterite) can be adjusted as a result of applied temperature or pH [39]. To Assess the apparent phase and structure of the formed polymorphs, X-ray diffraction was conducted within a range of 0-80 2θ° to identify the apparent peaks of the constitutes. The X-ray diffraction pattern is deployed in (Fig. 2c) remarking the crystalline nature of the structure. In this pattern, most of the three polymorphs peaks are indexed. The highest peak of intensity exists at 26. 12° for (111) plane indicating the orthorhombic aragonite (a=4.96 Å, b=8.0 Å, c=5.78 Å, and a space group P_{mcn} which matches former values [40–42]. Other standard peaks of aragonite are resident at 27.07° for (021), 32. 92° for (012), 36.15° for (200), 38.26° for (130), 41.11° for (211), 42.83° for (220), 45.74° for (221), 48.06° for (041) and 49.9° for (132) planes consecutively. These results coincide with prior reports with minor alterations [42,43], and the COD card no. 9015969 or JCPDS file No. 05-0453 [40,44]. while the superimposing of the peaks happens in some of the peaks. Less intense peaks for calcite exist in the pattern at 23.0° (012), 29.39° (104), 35.96° (110), and 39.40° (113) matching previous reports [42,43] and COD 9000095 or JCPDS card no. 83-0578 [44]. The calcite has a rhombohedral structure with a=b=4.9 Å, and c=17.06 Å with a space group R-3c [42]. Hexagonal vaterite with a space group of $P\text{-}6_{3/mmc}$ and a=b=4.13 Å, c=8.49 Å co-exists either. The peaks are located as 20.9° (002), 24.87° (100), 27.0° (101), 32.7° (102), 42.5° (004), 43.8° (210), 49.9° (104), and 55.73° (202) matching COD 9007475 or JCPDS no. 25-0127 [13,44]. The d-spacing of aragonite are 3.4 Å for (111) plane, 3.4 Å (021), 2.74 Å (012), 2.4 Å (200), 2.35 Å (130), 2.19 Å (211), 2.10 Å (220), 1.98 (221), 1.89 Å (041), 1.82 Å (132) . The d-spacing are implemented in the graph. Similarly, d-spacing for the vaterite and calcite are introduced. Vaterite 4.2 Å (002), 3.57 Å (100), 3.29 Å (101), 2.73 Å (102), 2.12 Å (004), 2.06 Å (210), 1.82 Å (104), 1.64 Å (202). Finally, the calcite 3.8 Å (012), 3.03 Å (104), 2.49 Å (110), and 2.28 Å (113). The quantification of the three combined mixed polymorphs can be conducted by the integrated intensities of the diffraction pattern peaks [42], while the peaks used are at (111), (104), and (101) for aragonite, calcite, and vaterite correspondingly. The determined intensities of the diffraction peaks per unit volume of calcium carbonate are approximately 0.697, 0.078, and 0.226 for Ia:Ic:Iv, which reflects the contributed portions of the three polymorphs. However, the emerged outcomes affirm most of the structure is aragonite. The zeta sizer for the particle size distribution is executed to determine the average size of the particles based on dynamic light scattering (DLS) as demonstrated in (Fig. 2d). The medium used for the preparation was water at room temperature. The hydrodynamic diameter [45] of synthesized polymorphs ranges from 10 nm to 100 nm which is densely located (d_{50}) at 23.1 nm.

3.2 Polymorphs-based gelling agent proposed application

Hydraulic fracturing fluids (HFF) are in great demand all over the world including the US particularly [46]. The selection and delivery of suitable hydraulic fracturing substances are crucial, therefore, the progress and evolution of this area of the application relies on scientists' and engineers' efforts at the most. The HFF employment causes potential risks such as freshwater consumption and water pollution. Hitherto, the water consumed in vertical wells for low-volume hydraulic fracturing ranges from 20k-80k gallons [46]. Furthermore, these amounts reach 3M-5M gallons per well in the Marcellus shale for high-volume hydraulic fracturing and may exceed 7M gallons for a multi-stage process [46,47]. Along with the provided amount of water, we need to add a specific ratio of the gelling agent. This ratio is around 10-1000 mg per liter [46]. Like so, the required cost for these huge quantities of supplied water and gelling agent based on Xantham gum for example after a few calculations (1gal=3.78-liter, 1 ton of Xantham gum= 7k $) may exceed 130k $ per fracturing single job (For more details on the expected cost check Table 1 Supporting information). In this section, the proposed application of the gelling agent in the area of hydraulic fracturing is investigated. The importance of the gelling agent as introduced earlier converges with multiple industrial sectors such as petroleum companies, cosmetics, and medical applications [48–51]. After acquiring the polymorphs and the successful preparation of the gelling agent, the agent was named (GA-CP22^{TM}

gelling agent) for potential commercial purposes. The materials safety data sheet (MSDS) of the gelling agent was introduced by the North Africa Mining and Petroleum Service Company (namc), covering the necessary specifications of the gelling agent following international standards. The Material safety data sheet (MSDS) of the agent is introduced in (Fig. 1 supporting information). The intended gelling agent offers a potential technique suitable for gelling naturally based polymorphs carbonates. One possible inquiry might come to mind on why we selected these initial constituents for the gelling agent (i.e., the calcium carbonates and the PEG). On the one side, the majority Xantham gum-based gelling agents require the insertion of other biocides additives to prevent the growth of fungus and bacteria that arise as a result of natural polysaccharides existence in Xanthan gum, while on the other side, the calcium carbonates minerals can buffer the pH of the solution offering prevention of the clay dissolution and subsequently impacting most reactions for stability, dissolution, porosity, and components stability in the reservoir [17,28,52,53]. Besides, the carbonates could scavenge the reactive oxidizing species (ROS) [54]. Inherently, the PEG is reported in wastewater and water treatment applications due to their preferred implications [55]. In short, PEG was used as surfactants, and anti-sedimentation agent where the molecular chain of the PEG provides a barrier shield preventing any occurring agglomeration that opposes the HFF process. Therefore, the combination of both Calcium carbonates and PEG is expected to be a desirable composite for the HFF production process.

The basic physical and chemical characteristics of the gelling agent are provided in the supporting materials (Fig. 1 supporting information). The initial assessment of the agent is provided in this work, further work will incorporate rheologaical, mechanical, and other parameters study such as temperature variation, pH value effect, and pressure.

Hydraulic fracturing is an industrial process conducted to seize natural gas, as well as oil from reservoirs owing to increasing suspension and transport inside the developed fractures. This process has evolved as a practice with higher importance in recent years [19,56] As reported in previous research, there are few undesirable side effects for the gelling agents for hydraulic fracturing fluids [57,58] The challenge is to overcome these obstacles which hinder better applicability and processing of the agent directed towards the hydraulic fracturing areas. The viscosity of the agent is demanded to facilitate the delivery of the proppant sand. The proppants maintain the open fractures enabling higher oil well productivity [21,59] Additionally, the insertion of the friction reducers with the gelling agent is also required [28]. Most of the familiar and commercial gelling agents rely on the polysaccharides such as Xanthan gum, and Guar gum. The anaerobic bacteria formation might occur which hinders the production casing, thus the addition of biocides is required to suppress the bacteria formation in the reservoir and water [60]. Following the major demands for the agent, the proposed agent in our report has the advantage of being non-toxic, as it is based on naturally existing abundant material, easy to synthesize, and cost-effective which is much less as reported in Table 1 and Table 1 Supporting information.

Furthermore, it is eco-friendly and doesn’t require the addition of oxidative agents such as ammonium persulphate to remove the free radicals. Unlike the familiar based Xanthma gum gelling agents the propsed agent doesn’t need the addition of urea to maintain basic nature of the media [19] as this potential agent relies on natural calcium carbonates known for their basic nature. Noteworthy to mention that, the employment of fossil diesel in the formation of fracturing concentrations promotes the emission of hurtful carbon dioxide which subsequently impacts global warming [61]. The agent is stable at high temperatures as well as it does not react with the surrounding media. Fig. 3. represents a photocopy of the prepared gelling agent. In the future, the authors will explore in depth the other specifications of the gelling agent. The much-reduced cost of the gel might propose its applicability on a wide scale for the forthcoming application.

Table 1 The comparison of the cost per single job for hydraulic fracturing fluids using around 5 M gallons of water, with the needed ratio of 1g of gelling agent for 1 L of water. Further detailed calculations are presented in Table 1 Supporting information.

The gelling agent based on Xantham Gum	The proposed gelling agent in our work
133k $	23k $

4.CONCLUSION

Rapid acquisition of the calcium carbonate polymorphs (i.e. aragonite, vaterite, and calcite) was provided in this work. The process included the function of naturally abundant coralline limestones in the Red Sea Coast as a source for the calcium carbonate polymorphs. The particle size and morphology of the carbonates were investigated through XRD, Particle sizer, and SEM. The results elucidated the side-by-side existence of the three mentioned polymorphs affirming preparation route validity. The function of the carbonates for the preparation of the gelling agent is performed in the work while the agent showed decent physical and viscous specification as well as much lower cost in comparison with Xantham gum-based gelling agents. The proposed gelling agent might be beneficial for different industrial applications such as polymers and the petroleum industry.

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