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Performance evaluation of bio/chemical leaching solution and nano catalyst in upgrading and enhancing oil recovery

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ABSTRACT

Heavy oil high viscosity poses significant challenges for recovery, with thermal methods often incurring high economic and environmental costs. Metal catalysts are known for their efficiency and effectiveness in oil upgrading processes. This research is novel as it emphasizes the utility of solid waste in catalyst preparation and introduces a groundbreaking comparative analysis of catalysts produced through both chemical and biological leaching methods, highlighting their environmental benefits and efficacy in improving oil recovery. We investigated the effects of various parameters influencing this process, including catalyst type, concentration, and reaction temperature, using design of experiments methodologies. The recovery of metal from electric arc furnace dust during chemical leaching was 15.1 %, while it was only 3.2 % in biological leaching. Additionally, the viscosity of the oil decreased by 10.2 %, 12.5 %, and 22 % with the addition of the bioleaching solution (conc: 0.01 wt.%, T: 50 °C), electric arc furnace dust (conc: 0.1 wt.%, T: 70 °C), and Fe_3O_4 (conc: 0.1 wt.%, T: 70 °C) nanoparticles, respectively. The reason for this is the catalytic activity of these metals, which facilitates the breakdown of heavy molecules and enhances the quality of crude oil. Notably, the enhancement in oil recovery after the upgrading process with the injection solution containing nano Fe_3O_4 and the biological catalyst was 61 % and 41 %, respectively. The comprehensive evaluation of catalytic performance under dynamic conditions provides valuable insights into optimizing recovery processes and underscores the dual economic and environmental benefits of this approach.

1. Introduction

Despite the increasing demand for renewable fuels, most of the world's energy is still supplied from fossil fuel sources. The amount of oil needed by the world in 2040 is expected to reach more than 111 million barrels per day, which is about 11 % more than the current oil consumption [1]. Compared to fossil fuels, renewable energy sources possess a number of disadvantages with regard to energy density, intermittency, and the challenges of energy storage. For example, in contrast to renewable sources, the output of energy from fossil fuels is steady and high density, while solar and wind depend on environmental conditions; thus, they produce variable energies. With the pre-existing infrastructure already in place, and lower upfront costs for fossil fuels, their greater energy density provides greater reliability in high demand and continuous energy supply [2–4]. Additionally, due to limited light oil reserves, the exploration, development, and production of heavy and extra-heavy oil from unconventional reservoirs are inevitable. Unconventional oil reservoirs include resources such as oil sands, extra-heavy oil, and certain shale oils. Furthermore, many reports indicate that unconventional resources comprise a notable portion of total oil reserves [5,6]. Compared to light oil reservoirs, production from heavy oil reservoirs faces challenges associated with the characteristics of heavy oil, such as high viscosity, a high carbon-to-hydrogen ratio, and many heteroatoms, including sulfur. Nitrogen, oxygen, and some metals such as nickel and vanadium are related [7,8]. Traditional thermal methods, such as steam injection, are commonly employed for reducing heavy oil viscosity and enhancing recovery. However, these methods often entail

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Nomenclature

λ	X-ray wavelength
β_d	peak width at half maximum
θ	diffraction angle
D	crystallite size
k	shape factor
μ	viscosity
m_i and m_f	mass of oil before and after exposure to temperature

high costs and environmental impacts, such as elevated CO_2 emissions from methane combustion. Consequently, innovative approaches like catalytic upgrading have emerged as sustainable and cost-effective alternatives, offering both enhanced oil recovery (EOR) and lower ecological footprints [9]. This oil has two main characteristics: high viscosity and low quality, and it is usually produced through steam injection. Still, the thermal efficiency of this process is low, so today; other methods are being developed for more productivity of oil resources. Catalytic methods of aquathermolysis are based on the injection of catalysts and catalysts along with steam into the stable formation, which can result in oil breakdown, viscosity reduction, oil mobility increase, and quality improvement [10]. Chemical reactions are an inseparable part of any process. In the industrial processes of oil recovery and heavy oil upgrading, catalysts are usually used to accelerate the reaction or increase efficiency. During these processes, chemical reactions with some oil components also occur, which causes changes in the characteristics and components of the oil structure. This process has been prescribed aquathermolysis [11]. A suitable catalyst could significantly facilitate the chemical reactions of heavy oil and reduce the viscosity effectively and irreversibly. Therefore, the catalysts participating in the in-situ reactions in the reservoir improve the quality of oil in the reservoir and enhance oil recovery [12]. Also, nanoparticles are studied as nano-catalysts in increasing the extraction of heavy oil and the combination of nano-technology with catalysts causes nano-catalysts to be used as markers of the reaction path and reaction accelerators that are not consumed during the reaction [13]. Oil recovery by in-situ and catalytic upgrading methods should be taken into consideration. Undoubtedly, oil viscosity reduction is the best indication of enhancing oil recovery by catalytic and in-situ methods [14]. Increasing oil recovery by catalytic and in-situ methods can be considered a cost-effective method with a high recovery rate. Also, this method enhances oil recovery simultaneously with upgrading oil, making the process economically feasible. By carrying out chemical reactions in the reservoir, heavy hydrocarbons can be converted into lighter hydrocarbons [15,16]. While ex-situ and in-situ upgrading methods share similarities, petroleum refinery processes utilize a well-designed reactor, whereas in-situ upgrading technologies rely on the reservoir acting as a natural reactor. Monitoring and regulating reservoir conditions, specifically temperature and pressure, during in-situ upgrading presents a challenging endeavor. It has been acknowledged that compared to other in-situ upgrading technologies, methods involving catalysis necessitate only the incorporation of catalyst addition devices, presenting a more straightforward approach. This method is considered appealing to decrease the viscosity of crude oil to levels suitable for transportation, thereby potentially enhancing the recovery factor [17].

In the pursuit of enhanced efficiency and eco-friendly techniques for extracting heavy oil, the introduction of bio-leached base catalysis represents a notable advancement. The application of catalysts in bioleaching solutions for in-situ upgrading of heavy crude oils introduces a new methodology that has garnered attention within the petroleum sector. With the ability to modify the viscosity and flow properties of heavy oil reservoirs, these biologically sourced agents have the potential to revolutionize extraction processes, leading to improved effectiveness and reduced environmental impact [18,19]. In general, bio-leaching means the use of microorganisms to dissolve and extract elements. Microorganisms that produce inorganic acids or organic ones can play an important role in recycling heavy metals from industrial solid wastes. *Aicidithiobacillus thiooxidans* is very well known in this group which is able to produce sulfuric acid from elemental sulfur. On the other hand some yeasts, for example, *Yarrowia lipolitica* can produce organic acid such as citric acid from cheap carbon sources [20,21]. The direct biological leaching process involves the presence of microorganisms. When waste is added at the beginning and during inoculation, the process is referred to as single-stage bioleaching. Two-step bioleaching is a process in which the waste is injected during the growth phase or at the beginning of the stationary phase when metabolites have been produced. Indirect bioleaching is a two-step process in which metabolites are separated and used to leach metal elements in the absence of microorganisms. Both chemical leaching and bioleaching are methods used to extract metals from various materials, including solid waste. The sources highlight the different aspects of these methods in terms of their efficiency and environmental impact. Chemical leaching often relies on the use of strong acids and other chemicals, which can contribute to environmental pollution if not properly managed. A key advantage of bioleaching is its potential for lower environmental impact compared to chemical leaching. Bioleaching is considered a "greener'' technology due to its reduced reliance on harsh chemicals and lower energy requirements [22].

Li et al. mentioned in their study that catalysts based on homogeneous transition metals, such as water-soluble inorganic salts, organic coordination compounds, and ionic liquids, can facilitate uniform interaction with oil phases, leading to improved viscosity reduction performance [23]. Thermally enhanced oil recovery methods, while effective in reducing the viscosity of medium-heavy crude oil, face significant challenges such as the loss of lighter hydrocarbons and coke formation. As a result, there is a growing preference for chemical-enhanced oil recovery (EOR) techniques, which, despite their potential environmental harm and generally lower recovery rates, can effectively increase oil recovery depending on reservoir characteristics. Strategies such as polymer and surfactant solutions have shown promising results, with studies indicating improvements of up to 55 % in oil recovery through optimized conditions. Chemical flooding techniques, particularly those using biopolymers like xanthan gum and gum Arabic, are increasingly favored for their environmental benefits and cost-effectiveness, with research illustrating a 49 % recovery enhancement when these materials are used with salt in high-viscosity reservoirs [24–27].

In EOR, a micromodel is a small-scale physical representation of a reservoir rock used in lab tests to investigate how fluids flow at the pore level. These models have been instrumental in uncovering the impacts of factors such as changes in wettability, the creation of microemulsions, and the role of salinity in enhancing oil recovery rates. By employing micromodels, scientists can study and interpret intricate processes happening at the pore level that are challenging to observe in larger and more intricate reservoirs [28–31].

Heavy and extra-heavy oil recovery faces significant challenges, including high viscosity, high carbon-to-hydrogen ratios, and the presence of heteroatoms. While catalytic aquathermolysis has shown promise for in-situ upgrading, there is limited research on using recycled industrial metal waste for catalyst preparation. The application of bioleaching in oil upgrading is underexplored, particularly in comparison to chemical leaching. Bioleaching offers environmental benefits and its comparative performance in oil recovery and upgrading remains poorly understood. Micromodel studies are essential for visualizing oil recovery processes, yet few have comprehensively analyzed the dynamic effects of catalysts (bioleaching, chemical leaching, and nanocatalysts) under controlled conditions.

This research is novel in several key aspects, highlighting the importance and efficiency of catalysts in enhancing oil recovery and

upgrading heavy oil. Additionally, the methodology shows potential for improving recovery from unconventional oil resources, offering a versatile approach to address the challenges associated with these reservoirs while safeguarding the environment and reducing the ecological impact of the processes by utilizing metal waste to prepare catalysts. For the first time, this study employs both chemical and biological leaching methods to produce catalysts aimed at improving oil recovery and upgrading heavy oil. Furthermore, it synthesizes an iron oxide metal nanocatalyst (Fe_3O_4) and represents the first comparative analysis of the performance and efficiency of these newly developed catalysts.

2. Material and methods

2.1. Materials

The oil used in this study was sourced from a field in southwestern Iran. It had a density of 0.9343 g/cm^3 (19.95 °API) and a viscosity of 295 centipoise (cp) at room temperature (25 °C), with an asphaltene content of 11.7 %. $FeCl_3{\cdot}6H_2O$, $FeCl_2{\cdot}4H_2O$, ammonia solution (NH_4OH), toluene, and methanol were prepared from Merck company. The culture medium consisting of the following components (g/l): 17.65 $Na_2HPO_4{\cdot}12H_2O$, 7 KH_2PO_4, 1.5 $MgSO_4{\cdot}7H_2O$, 0.2 $CaCl_2{\cdot}H_2O$, 0.02 $ZnSO_4{\cdot}7H_2O$, 0.06 $MnSO_4{\cdot}H_2O$, 0.15 $FeCl_3{\cdot}6H_2O$, and 0.27 yeast extract in neutral pH. The supplier of the electric arc furnace dust (EAFD), Tabarestan steel factory in Mazandaran, Iran, and *Yarrowia lipolytica* IBRC-M30168 was purchased from the Iranian Biological Resource Center (IBRC) in Tehran, Iran.

2.2. Biological leaching

Indirect bioleaching was employed to dissolve metals from EAFD. At the first stage, metabolite was produced in 80 g/l of crude sunflower oil, a cost-effective and hydrophobic carbon source, and during 9 days of fermentation at 140 rpm and 30 °C in mentioned culture medium. The yeast biomass was separated from the produced metabolites by centrifugation at 10,000 rpm for 20 min. Subsequently, the yeast-free culture medium was utilized to bioleaching the EAFD. Bioleaching experiments were conducted in Erlenmeyer flasks within a shaker incubator at 140 rpm and 60 °C in the presence of 50 g/l pulp density for 6 days. At the end of this stage, the bioleachate was separated from the residual EAFD using a centrifuge at 8000 rpm for 15 min and then subjected to an inductively coupled plasma (ICP) (Vista-Pro, Varian, Australia) analysis. More details can be found in [20,21]. This solution was subjected to use as a bio-leached base catalyst for upgrading experiments. The procedure for the bioleaching process can be observed in Fig. 1.

2.3. Chemical leaching

In order to prepare a chemical leaching solution from EAFD, 30 g powder was gradually mixed into a 300 mL solution of hydrochloric acid and nitric acid, with a ratio of 3:1, for 30 min at 120 °C. After boiling the powder in the mixture of two acids for 6 h (adding fresh acid to the container as needed), the solution is allowed to cool down. After passing the solution through filter paper, the dissolved part is brought to volume in a 500 mL flask. The prepared solution was subjected to ICP analysis in order to identify metal concentration. Test results of original EAFD,

Table 1
ICP result of original EAFD metal content.

	Metal content (g/kg)				
EAFD	Al	Zn	Cr	Mn	Fe
	5.38	10.4	14.4	41.5	274.1

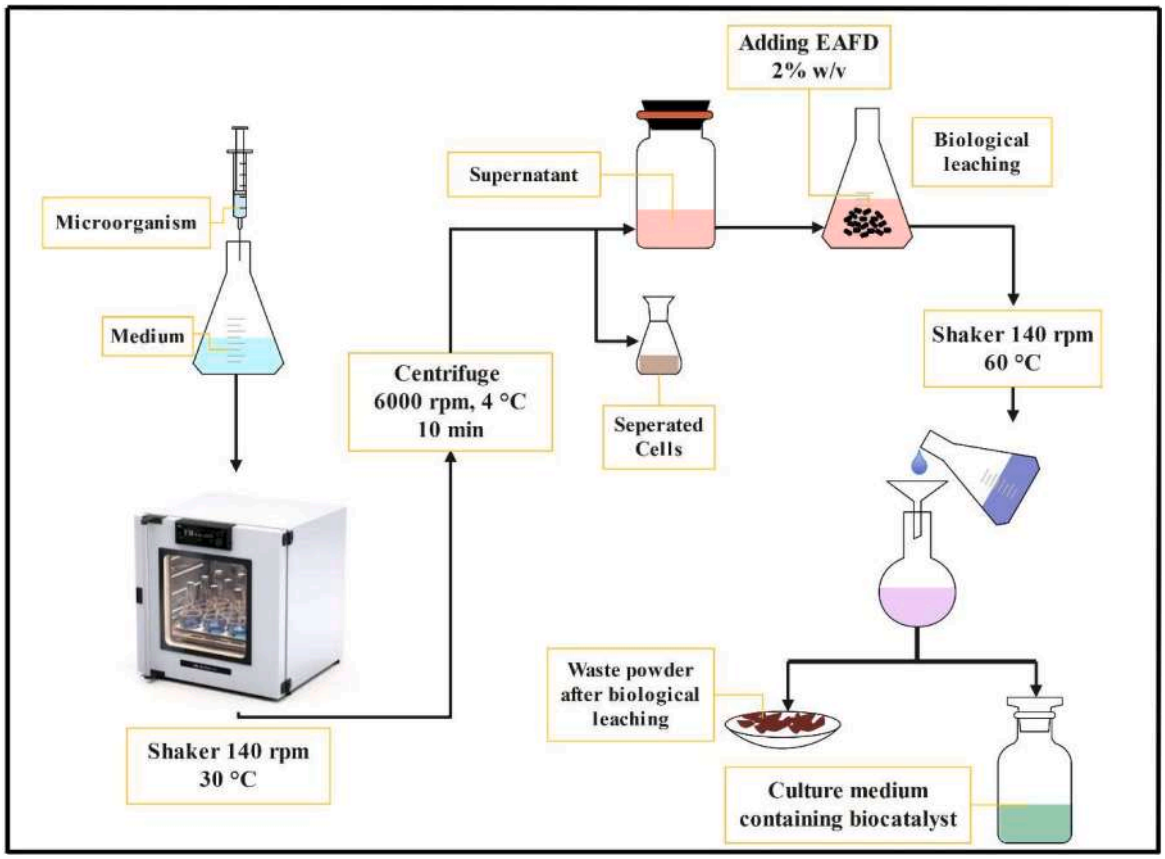

Fig. 1. Biological leaching process.

chemical, and biological leaching solutions are shown in Tables 1 and 2, respectively.

2.4. Synthesis of magnetic iron oxide (Fe_3O_4)

In order to synthesis magnetic iron oxide (Fe_3O_4), 3.08 g $FeCl_2{\cdot}4H_2O$ and 8.4 g $FeCl_3{\cdot}6H_2O$ and 400 mL of deionized water is combined in a three-hole flask, then the solution is placed in a water bath at 80 °C. The agent Precipitator is utilized with a 30 % ammonia solution to adjust pH and precipitate nanoparticles. Throughout the procedure, ammonia solution was incrementally introduced until the solution's pH level reached 11. Simultaneously, the application of nitrogen gas pressure while heating the solution also serves to prevent the reaction of iron oxide. At this point, a black Fe_3O_4 solid is produced. After the necessary time for the solution to settle, the nanoparticles that were produced were rinsed. Next, the black precipitate was dried in an oven at 70 °C for 6 h [13].

2.4.1. Characterization

Different assessments described the produced Fe_3O_4 nanoparticles and additional catalysts. Fourier transform infrared spectroscopy (FTIR) was utilized at room temperature to identify the functional groups on nanoparticles. Additionally, the Fe_3O_4 nanoparticle powder was subjected to X-ray diffraction (XRD) analysis.

2.4.1.1. FTIR. FTIR is a technique used to analyze chemical bonds and functional groups. It helps determine the presence, absence, or changes in bonds within a material. In this process, infrared (IR) light passes through a sample, where some is absorbed and some transmits. The resulting spectra reflect the sample's molecular characteristics based on IR photon absorption. For absorption to occur, the molecule's dipole moment must change during vibration. This allows the effective detection of functional groups and molecular structures. The data obtained enables the identification of specific functional groups and bonds in synthesized compounds. In this study, FTIR (PerkinElmer Spectrum Analyzer, Version 10.03.06, USA) was employed to analyze the functional groups present in dried powder samples of hybrid nanocatalysts at room temperature, within the range of 400–4000 cm^{-1}.

2.4.1.2. XRD

To quantitatively analyze XRD results, techniques like Scherrer and Williamson-Hall are utilized. The Williamson-Hall method was used to determine the crystallite size of the samples from the XRD (X'Pert MPD, Philips, Netherlands) data. Peak broadening in XRD is influenced by both crystallite size and strain within nanocrystals. Scherrer's equation (Eq. (1)) for calculating crystallite size involves the X-ray wavelength (λ = 0.154 nm for K_α radiation), the peak width at half maximum (β_d), the diffraction angle (θ), and the crystallite size (D). The shape factor (k) typically ranges from 0.9 to 1.

$$D = \frac{k\,\lambda}{\beta_d \cos\theta} \tag{1}$$

2.5. Effective parameters and design of experiment

To operationalize this research, the effect of influencing parameters on this process, such as the type and concentration of catalyst and reaction temperature, will be carefully investigated with the help of experimental design methods. In this regard, appropriate test design methods will be used to study accurately and reduce the required tests. The purpose of test design is to select the most critical variables and test conditions so that the process can be investigated in the best way through them, and at the same time, the number of tests is logical. Therefore, designing the appropriate number of experiments and conducting the necessary analyses using the existing practical statistical methods is necessary. These analyses are often done with the help of statistical analysis software (the Design-Expert package). At this stage, an experiment design table was designed by the General Factorial method. Then, each suggested implementation is done in the laboratory, and the results are presented for analysis and review. The factors being considered include the temperature, type, and concentration of the catalyst, with their values determined from previous studies and pretests, and the output responses, including the viscosity and mass loss of the oil sample. The level of each of these parameters is listed in Table 3.

The mass reduction percentage was computed using Eq. (2), where m_i and m_f represent the mass of oil before and after exposure to temperature. The purpose of measuring the change in sample mass before and after processing is to account for any mass loss, which may result from the evaporation of lighter oil components. This residual matter in the reservoir can function as a secondary gas cap, potentially displacing oil towards the production well.

$$\text{mass reduction} = \frac{m_i - m_f}{m_i} \times 100 \tag{2}$$

2.6. Dynamic tests

The graphic representation of the micromodel flooding system is shown in Fig. 2. This system includes a syringe pump, light source, computer, waste container, and powerful camera for taking pictures of the micromodel sample. The oil recovery factor was determined through photo analysis employing Photoshop tools before and after the flooding by visualization and comparisons of the pixels of crude oil (in black colors) and injection fluids (in light colors).

The porous medium was first designed in Corel Draw software, then this design was engraved on the glass using a laser. Then with a sandpaper, the micromodel has been smoothed and the necessary space for fluid flow has been provided. When the micromodel is placed in the furnace with a temperature of 700 °C, the two surfaces of the glass are stuck together and only the space of porous medium remains. Then since the porous medium was oil-wet, oil was injected into it. In the flooding operation, to obtain the recovery factor, the image was recorded by the camera with a time interval of one minute, and the amount of oil recovery was obtained by analyzing these images and counting the pixels of each phase. Table 4 shows the specifications of the micromodel. In addition, it is worth mentioning that in all processes, one pore volume fluid with a rate of 0.07 mL/min was adjusted as prevailing conditions for flooding tests. Also, all the injection tests were conducted at ambient pressure and temperature.

3. Result and discussion

In this section, the characterization tests are discussed first, followed by a detailed examination of the Design of experiments (DOE) results. The effects of the catalyst on oil samples are then investigated through FTIR analysis and dynamic testing in micromodel.

Table 2
ICP results of chemical and biological leaching solutions.

	Metal concentration (mg/l)				
Solution	Fe	Zn	Mn	Cr	Al
Chemical leaching	41.3	2.1	8.4	3.2	1.1
Bioleaching	9.1	13.8	12.2		

Table 3
Parameters and their levels.

Parameter	Level 1	Level 2	Level 3
Type of catalyst	Bio-leaching solution	EAFD	Fe_3O_4 nanoparticles
Temperature (°C)	50	70	90

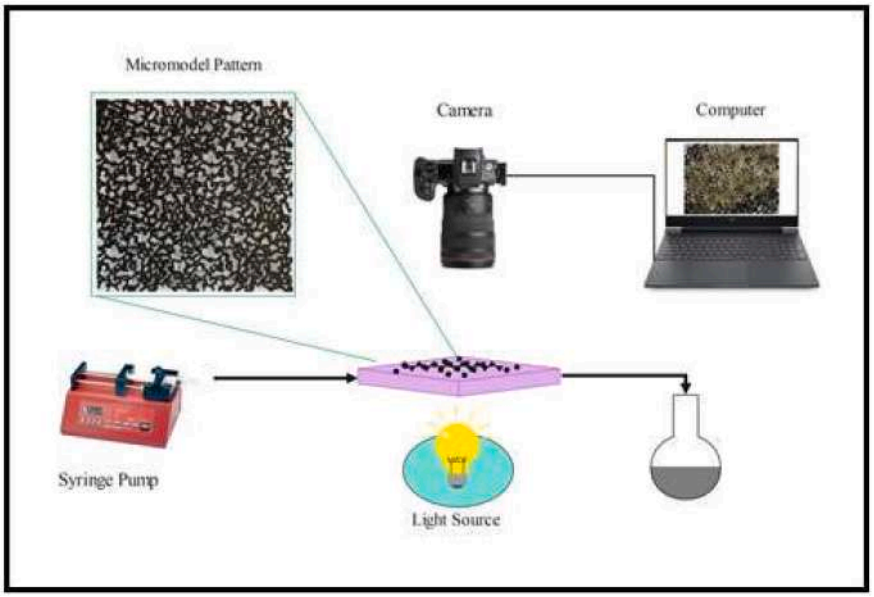

Fig. 2. Schematic of the micromodel flooding system.

Table 4
Specification of the micromodel.

Properties	Size (cm)	Thickness (μm)	Pore volume (cc)	Permeability (mD)	Porosity (%)
	6 × 6	110	0.146	840	38

3.1. FTIR analysis

FTIR analysis involved utilizing powdered solids dispersed in a KBr matrix to confirm the surface alteration of the nanoparticles produced. Fig. 3 displays the spectra of nanoparticles made of Fe_3O_4. Based on the IR spectra in Fig. 3. The Fe—O band in Fe_3O_4 nanoparticles is responsible for the peaks at 445 and 569 cm^{-1}. The band at 1343 cm^{-1} is attributed to the linkage of —OH bending vibration with Fe atom [30]. The peak associated with the stretching vibration of the O—H band falls within at 3370 cm^{-1}. The 3700 cm^{-1} peak corresponds to the unbound NH_2 group and overlaps with the O—H band [32,34].

3.2. XRD analysis

XRD analysis was conducted to assess the properties of the nanoparticles, including their character, texture, crystal orientation, and crystal size. The XRD pattern of Fe_3O_4 nanoparticles is illustrated in Fig. 4. According to JCPDS No. 00-003-0862, the analysis revealed distinct diffraction peaks at $2\theta = 30.1°$, 35.5°, 43.3°, 53.6°, 57.6°, and 62.3°. These peaks correspond to the crystalline planes (220), (311), (400), (422), (511), and (440) of Fe_3O_4 nanoparticles, respectively [33,34]. The synthesis of Fe_3O_4 nanoparticles is confirmed to be complete as shown in Fig. 4. X-ray analysis revealed the distribution of crystal sizes, and the XRD analysis estimated the crystallite size of Fe_3O_4 nanocatalysts to be about 12 nm. The average particle size of the synthesized Fe_3O_4 nanocatalysts is 25 nm [13].

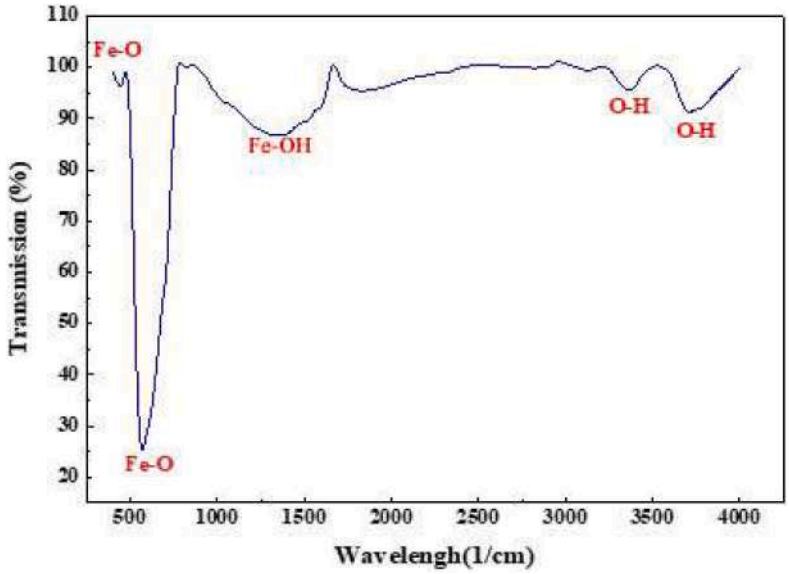

Fig. 3. FTIR pattern of synthesized Fe_3O_4 nanoparticles.

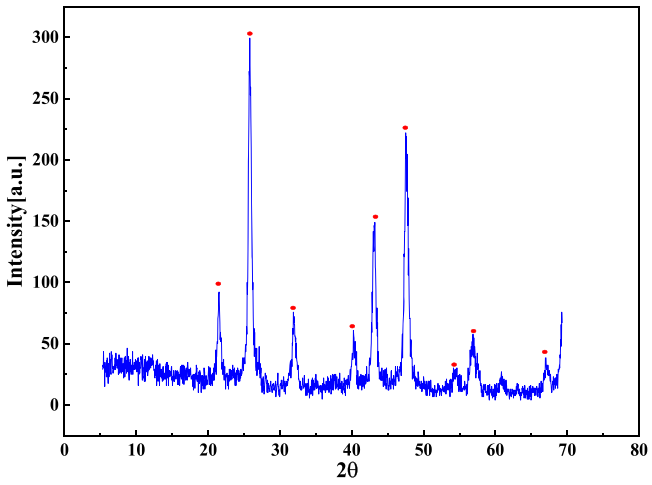

Fig. 4. XRD pattern of synthesized Fe_3O_4 nanoparticles.

3.3. DOE results

Table 5 presents the results of the DOE, which include the viscosity (cp) and the mass reduction (g) of the oil samples.

In the two-factor interaction (2FI) model, interactions between the factors are considered, but no higher-order effects occur, making the 2FI model the most suitable choice. Fig. 5a and b illustrate predicted vs actual values for viscosity and mass reduction respectively. The 2FI correlations show the effects of the factors (temperature and concentration) on the viscosity (cp) or mass reduction (g) for each catalyst. Table 6 displays the relationships between the actual factors and the responses. According to Table 6, higher temperature (T) and lower concentration (C) lead to degradation and evaporation of oil components, which results in higher viscosity (μ) and mass reduction percentage. Therefore, higher temperatures and lower concentrations are unfavorable for oil samples.

Statistical results indicate that the 2FI model aligns well with the experimental data, as demonstrated by high coefficients of determination (R^2 = 0.9368 and 0.9433 for viscosity and mass reduction, respectively). At this stage, 2FI correlations were utilized, and their performance in correlating factors and responses was evaluated. The analysis of variance (ANOVA) tables for each response are provided in Tables 7 and 8. Based on the ANOVA results, the p-values for both viscosity and mass reduction were <0.05, indicating that both are significant. Additionally, the F-values demonstrate that the type of catalyst and the temperature were the most effective parameters for viscosity and mass reduction, respectively.

Table 5
The result of DOE.

Run	Factors			Responses	
	A:Type	B:T (°C)	C: Concentration (wt.%)	Viscosity (cp)	Mass Diff (%)
1	Bio-leaching	50	0.10	280	0.11
2	Bio-leaching	90	0.10	440	0.85
3	Bio-Leaching	70	0.01	350	0.50
4	Bio-leaching	70	0.10	335	0.37
5	Bio-Leaching	90	0.01	368	0.64
6	Bio-leaching	50	0.01	265	0.02
7	Fe_3O_4	90	0.10	282	0.76
8	Fe_3O_4	50	0.01	260	0.05
9	Fe_3O_4	70	0.01	245	0.29
10	Fe_3O_4	70	0.10	230	0.32
11	Fe_3O_4	90	0.01	272	0.58
12	Fe_3O_4	50	0.10	250	0.24
13	EAFD	50	0.10	268	0.18
14	EAFD	50	0.01	278	0.11
15	EAFD	70	0.10	258	0.34
16	EAFD	70	0.01	270	0.42
17	EAFD	90	0.10	280	0.81
18	EAFD	90	0.01	282	0.69

Fig. 6 illustrates how the viscosity of oil solutions with different catalysts varies with changes in temperature and concentration. Fig. 6a indicates that the sample containing Fe_3O_4 nanoparticles has the most significant impact on reducing oil viscosity, with a reduction ranging from 10.2 % to 17.5 %, depending on temperature and concentration. The reason for the decrease in viscosity is the increase in the stability of asphaltene particles through an electrostatic mechanism. Metal particles and nanoparticles, due to their surface charge, keep asphaltene particles in suspension and stabilize them, because aggregation of heavy molecules such as asphaltene can increase the viscosity of oil. These metallic catalysts can also reduce the activation energy of the breakdown of heavy oil molecules. Therefore, heavy crude oil molecules are broken and converted into lighter molecules, which have a lower viscosity. In addition to the stabilization of asphaltenes, metallic catalysts serve several crucial functions during the upgrading process. Firstly, their high surface area allows for enhanced interaction with heavy hydrocarbons, promoting efficient catalytic reactions. The catalytic activity can facilitate various reactions, including hydrocracking, deasphalting, which collectively promote the formation of lighter hydrocarbon products. Moreover, it is evident that the viscosity decreases as the temperature decreases and concentration increases. The viscosity reduction with EAFD is less pronounced than with Fe_3O_4 nanoparticles, ranging from 6 % to 10.5 %. However, Fig. 6b indicates that at high temperatures and concentrations, the oil viscosity increases significantly for the leaching solution, likely due to interactions between the oil and the bio-leaching solution. In contrast, the solution with steel furnace dust, as shown in Fig. 6c, exhibits minimal variation in viscosity with changes in temperature and concentration.

As shown in Fig. 7, the mass reduction of samples in all cases was less than 0.7 %, which can be considered negligible. Fig. 7a demonstrates the mass reduction of the oil samples as a function of temperature and concentration of Fe_3O_4 nanoparticles. Fig. 7b shows that with EAFD as the catalyst, mass reduction increases with higher temperatures and concentrations. However, the amount of mass reduction is less than with the Fe_3O_4 nanoparticle catalyst. Fig. 7c indicates that with the bio-leaching solution, the mass reduction also increases with higher temperatures and concentrations. Fig. 7 indicates that higher temperature and concentration increase the mass loss for all samples. This is caused by the cracking of heavy hydrocarbon chains into smaller molecules and the volatilization of small molecules from the solution. The sample with Fe_3O_4 nanoparticles showed the most mass loss among the three samples, as it has the most optimal catalytic activity and can break large chains more effectively.

The main objectives of DOE in this research were to minimize the viscosity and value of oil mass reduction. The maximum, minimum, and optimum factors, corresponding responses, and target of each response are shown in Table 9. Due to desirability, this condition reduced the viscosity to 264, and the value of mass reduction was equal to 0.124.

3.4. FTIR analysis of oil samples

The infrared spectrum shows the amount of absorption at different wave numbers, which correspond to specific chemical bonds. Therefore, the wave number of each peak reveals the presence of a certain functional group in the sample. In this analysis, the peaks are interpreted to identify and compare the functional groups and the structure of oil samples with different catalysts. Catalysts provide an alternative reaction pathway with a lower activation energy, thus speeding up the reaction rate at lower temperatures and promoting the breaking of carbon-hydrogen bonds, which is essential for hydrocracking reactions.

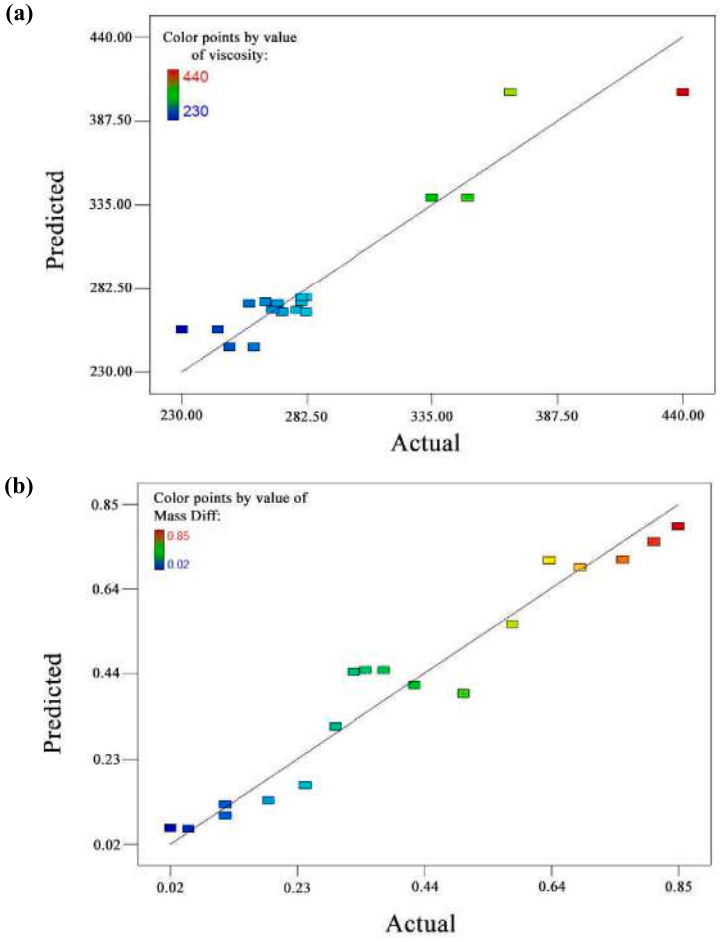

Fig. 5. Predicted vs actual values for a) Viscosity and b) Mass reduction.

Table 6
Correlations of viscosity and mass reduction for each catalyst.

Catalyst	Correlations
Fe_3O_4 nanoparticles	$\mu = +251.35 + 0.12T - 606.48C + 7.87TC$
	Mass reduction $= -0.57 + 0.01T + 0.4C + 0.01TC$
EAFD	$\mu = +293.85 - 0.23T + 639.81C + 7.87TC$
	Mass reduction $= -0.60 + 0.01T - 0.63C + 0.01TC$
Bio-leaching solution	$\mu = +125.18 + 2.85T - 284.26C + 7.87TC$
	Mass reduction $= -0.75 + 0.016T - 0.41C + 0.01TC$

Table 7
ANOVA table for the viscosity response.

Source	*p*-value	*F*-value	Mean square	Sum of squares	Df
Model	0.0007	13.17	4752.66	42,773.94	9
A-TYPE	0.0001	32.34	11,667.06	23,334.11	2
B-T	0.0012	24.1	8694.08	8694.08	1
C-Conc.	0.6929	0.17	60.5	60.5	1
AB	0.0033	12.68	4573.08	9146.17	2
AC	0.3248	1.3	468.5	937	2
BC	0.2325	1.67	602.08	602.08	1

The FTIR test examines the infrared spectrum of a sample to identify chemical bonds and functional groups by absorption or emission. The peaks corresponding to asymmetric and symmetric CH_2 bonds are observed at 2850 cm^{-1} and 2926 cm^{-1}, respectively. The CH_3 bonds are depicted by the peaks at 2962 cm^{-1} and 2872 cm^{-1}, with one being asymmetric and the other symmetric. The CH_3 umbrella peak appears at

Table 8
ANOVA table for the mass reduction.

Source	*p*-value	*F*-value	Mean square	Sum of squares	Df
Model	0.0004	15.07	0.1276	1.15	9
A-TYPE	0.6068	0.5320	0.0045	0.0090	2
B-T	<0.0001	128.95	1.09	1.09	1
C-Conc.	0.1197	3.03	0.0257	0.0257	1
AB	0.5204	0.7095	0.0060	0.0120	2
AC	0.6463	0.4612	0.0039	0.0078	2
BC	0.6293	0.2519	0.0021	0.0021	1

1375 cm^{-1}, along with CH_2 rocking at 1455 cm^{-1} and at 720 cm^{-1} peaks [35–37]. The peaks correspond to the hydrocarbon composition of the oil, while the peaks from 400 cm^{-1} to 1000 cm^{-1} are associated with the presence of aromatic structures in the oil. Hence, the oil sample includes hydrocarbon structures that are both linear and aromatic. The presence of polar functional groups in asphaltenes induces surface activity, leading to van der Waals and electrostatic attractions between asphaltene aggregates and nanoparticle surface sites. The nanoparticles, therefore, act as sites for asphaltene adsorption, which is a key step in fuel formation.

Fig. 8 shows the FTIR spectra of five oil samples. The samples treated with chemical and bio-leaching catalysts have higher peak intensities for CH_2 and CH_3 vibrations, indicating an increase in alkanes and an upgrade in crude oil quality. The bio-leaching catalyst-treated samples show a more pronounced peak enhancement, suggesting a more effective upgrading process. Additionally, the CH_3 peaks increase more significantly than the CH_2 peaks, indicating a preferential formation of

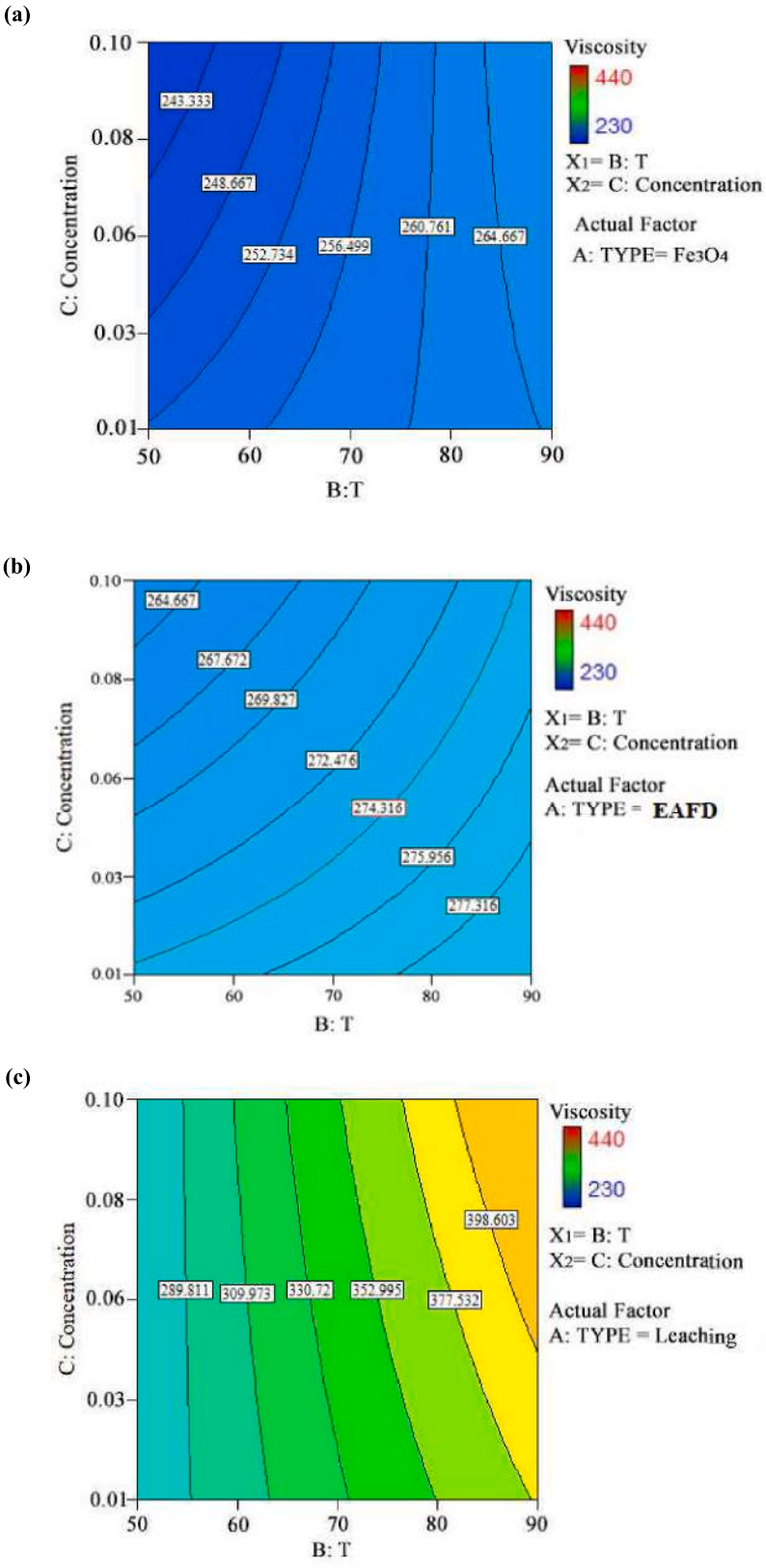

Fig. 6. The effect of temperature-concentration interaction with a) Fe_3O_4 nanoparticle, b) EAFD and c) Bio-leaching and on viscosity.

lighter hydrocarbons during the upgrading process. Regarding the aromatic compounds present in the oil, notable peaks associated with cyclic C═C (1400–1620 cm^{-1}), neutral C—H (1000–1200 cm^{-1}), and the out-of-plane bending modes (700–1000 cm^{-1}) indicate the presence of aromatic structures. However, we observed that the intensity of the aromatic peaks in the oil samples treated with Fe_3O_4 nanoparticles and steel furnace dust powder is lower compared to the other three samples. Redox cycle involves the change of the oxidation state of the metal

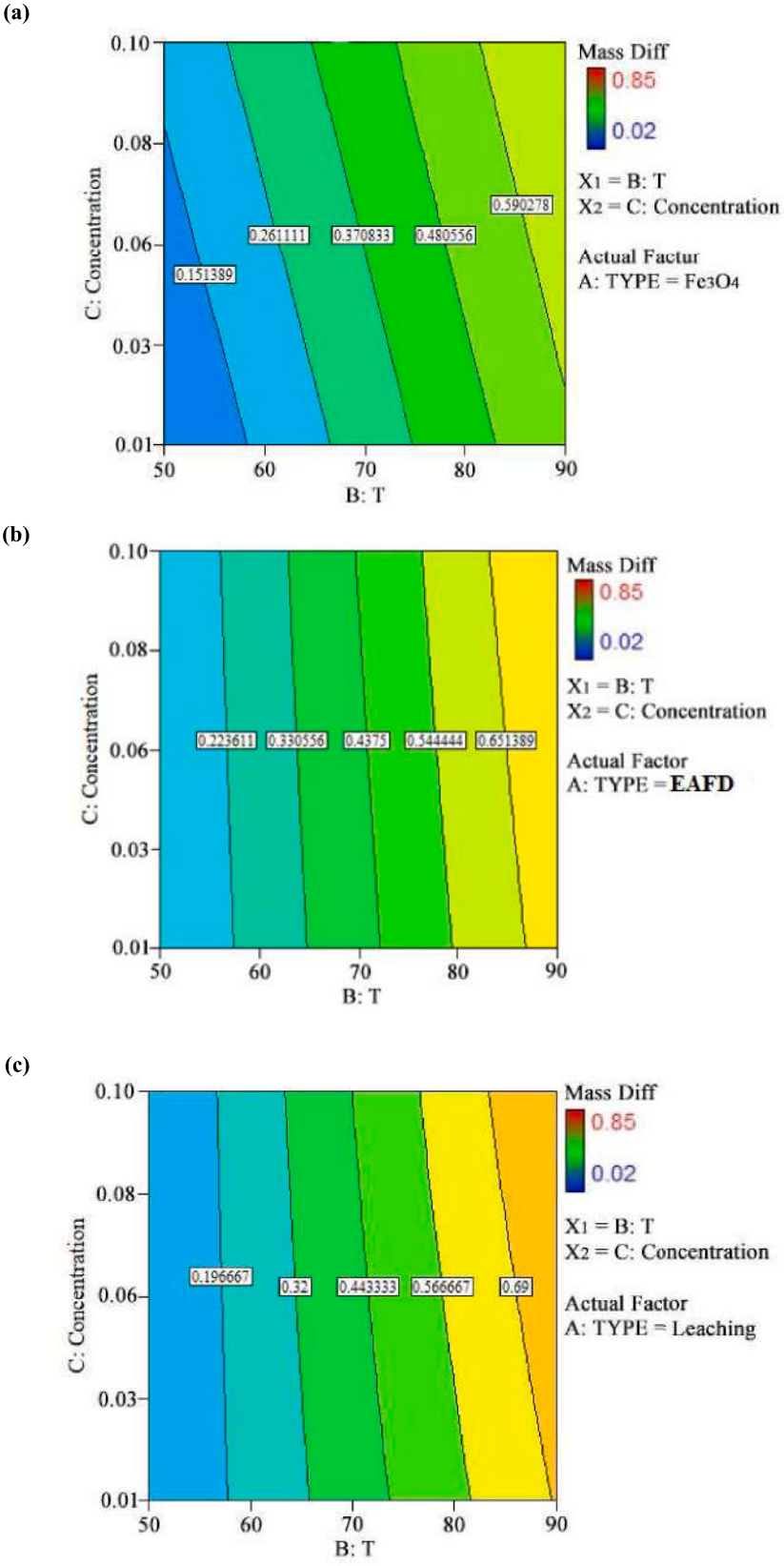

Fig. 7. The effect of temperature-concentration interaction with a) Fe_3O_4 nanoparticle, b) EAFD and c) Bio-leaching on mass reduction.

Table 9
Optimization criteria and optimum values for factors and corresponding responses.

Response	Name	Min.	Max.	Objective	Importance
Y1	Viscosity	230	440	minimize	+++++
Y2	Mass reduction	0.02	0.85	minimize	+++++
Optimum values					
Type of catalyst	Concentration	Temperature	Viscosity	Mass reduction	
Fe_3O_4 nanoparticle	0.07	50	264	0.124	

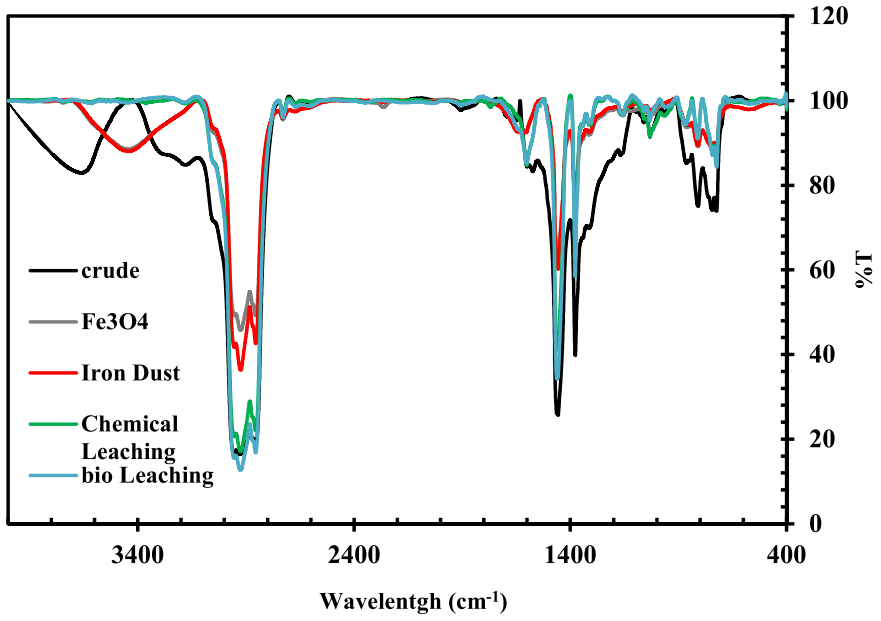

Fig. 8. FTIR analysis of crude oil and oil containing various catalysts.

cations with the release/bonding of surface oxygen. During coke oxidation, oxygen from the metal oxide surface reacts with coke to form labile CO_2 as carbonates on the surface. The decomposition of these carbonates is then fueled by gas-phase oxygen, which causes the re-oxidation of the metal oxide. This reduction suggests that these catalysts are effective in diminishing the concentration of aromatic compounds in crude oil. Moreover, the aromatic peaks in the samples treated with chemical and bio-leaching catalysts are also lower than those found in the untreated crude oil, indicating that these catalysts facilitate the transformation or reduction of aromatic compounds, contributing to a more desirable alteration of the crude oil composition.

3.5. The process of EOR in the dynamic system

The injection in the micromodel has been done in such a way that the initial injection was done by the fluid until the moment of breakthrough time. After that, the micromodel is placed in the oven at the optimal temperature and time. Then, in order to recover the upgraded oil, fluid was injected again in the amount of a Pore volume. According to the results obtained from the static tests, the optimal temperature and time for the upgrading process was 50 °C and 6 h. At this stage, in total, the amount of EOR by 4 fluids has been investigated, including bio-leaching solution, chemical leaching solution, the solution containing Fe_3O_4 catalyst, and water, the results of which are presented below.

3.5.1. Investigating the oil recovery factor with different catalysts

Fig. 9a and b show the amount of oil recovery in water injection, before and after the upgrading process. As shown, before the upgrading process, the amount of oil recovery was 17 %. Water injection has caused viscous fingering to occur, preferably penetrating the less-resistant areas in the porous media and leaving pockets of oil trapped. This effect greatly reduces the ultimate oil recovery factor. After being placed in the oven for 6 h at a temperature of 50 °C, this amount has increased to 26 %, This increase is due to the effect of temperature on the viscosity of oil, which has reduced its viscosity. Fig. 9c and d show the amount of oil recovery in the injection of biological leaching solution with a concentration of 0.01 wt.%, before and after the upgrading process. Before the upgrading process, the oil recovery rate was 30 %, after being placed in the oven, this amount increased to 41 %, which is due to the catalytic effect of the biological leaching solution on the oil and its upgrading, which Finally it has led to increased oil recovery. It is worth mentioning that, after the bioleaching process, the catalysts are completely solution in the base fluid, without any sediment. This phenomenon is attributed to the fact that in the bioleaching process, insoluble metal sulfides change to the solution metal sulfates [34,35]. Thus, this cannot lead to any drawbacks during the injection process in the micromodel.

Fig. 9e and f show the amount of oil recovery in the injection of a solution containing Fe_3O_4 nano-catalyst with a concentration of 0.1 wt. %, before and after the upgrading process. Indeed, in the micromodel, fluid primarily flows through the pore spaces, and the displacement efficiency is constrained by capillary forces that trap oil within the pore corners. Therefore, the presence of nanoparticles allows the displacing fluid to bypass areas of low permeability, effectively reducing the impact of capillary forces in those regions. Before the upgrading process, the oil recovery factor was 44 %, after placing it in the oven and performing the upgrading process, this amount increased to 61 %, this increase is due to the catalytic effect of the solution containing Fe_3O_4 on oil and the significant decrease in its viscosity which has been fully evident in static tests and has finally led to an increase in oil recovery. Fig. 9g shows the

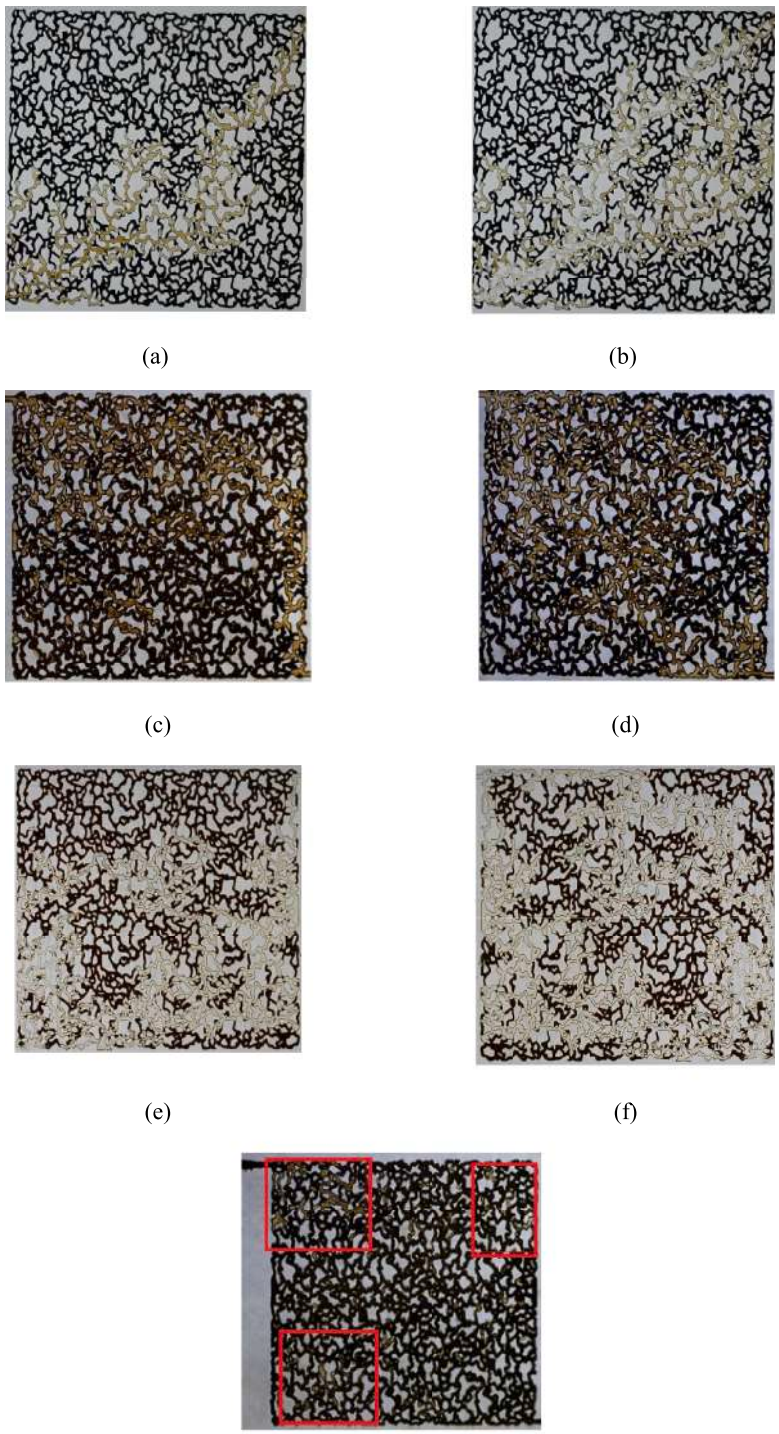

Fig. 9. Micromodel flooding with water injection, (a): Before upgrading. (b): After upgrading; Bio-leaching solution (c): Before upgrading. (d): After upgrading; solution containing Fe_3O_4 nano-catalyst, (e) Before upgrading. (f) After upgrading and (g) Chemical leaching solution in the micromodel containing formation of sludge.

injection of chemical leaching solution in the micromodel. As it is clear in the figure, due to the presence of a high percentage of asphaltene in the oil and the presence of a percentage of acid in the chemical leaching solution and then the formation of sediment in the collision of acid and asphaltene, the injection and calculation of recovery will face an error. In the figure, the creation of slug flow and then the return of the flow in the path is visible. For this reason, the recovery percentage cannot be calculated. This test has been repeated three times, and the mentioned incident has been observed all three times.

Fig. 10 presents the recovery factor results for heavy oil. The results illustrate an increase in oil recovery when catalysts are employed. Additionally, heating the oil and injecting fluid in an oven further increased recovery a result attributed to the interaction between oil and catalysts leading to viscosity reduction and oil upgrading, as observed in static tests.

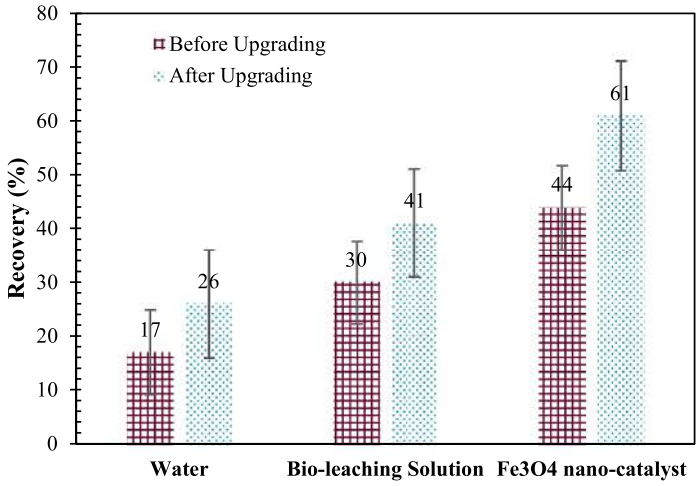

Fig. 10. Comparison of recovery factor during injection of different catalysts into micromodel.

3.6. Environmental and economic implications

One of the standout aspects of this research is the use of recycled metal waste to prepare catalysts, particularly through biological leaching methods. This approach not only provides a cost-effective source of catalytic materials but also addresses significant environmental concerns associated with industrial waste. By diverting metal waste from disposal and reintroducing it into the production cycle, the study contributes to reducing the environmental footprint of oil extraction and processing operations.

The biological leaching method demonstrated lower metal recovery efficiency compared to chemical leaching (3.2 % vs. 15.1 %). In fact, chemical leaching using acids or complexing agents is an effective method for removing heavy metals. However, this approach has a significant downside. For instance, it often requires a large amount of chemicals and is only economically viable at high plant capacities. Conversely, bioleaching presents a feasible and sustainable alternative that can lower the costs associated with chemical leaching. This process relies on microorganisms that produce bio-acids, which solubilize heavy metals. Specifically, bioleaching is facilitated by certain acidophilic bacteria that oxidize reduced sulfur or ferrous iron, thereby generating acidity [38,39]. As a result, bioleaching not only reduces the use of chemicals but also minimizes environmental impact, making it a more sustainable option. This trade-off between efficiency and environmental sustainability is a key consideration for future research and industrial application. The study suggests that further optimization of biological leaching processes could enhance metal recovery rates, making it a more competitive option against traditional chemical methods. Future prospects for this research involve a thorough examination of parameters such as injection rate, well patterns, and the number of injection wells. Utilizing both numerical and experimental methods will provide valuable insights into optimizing these factors, thereby facilitating the operationalization of this upgrading technique. Future studies should focus on replicating high-pressure and high-temperature reservoir conditions and conducting pilot-scale trials to assess the scalability and practical applicability of the catalysts. Additionally, integrating recycled catalysts with advanced EOR techniques, such as polymer or surfactant flooding, could further optimize oil recovery and promote sustainable practices.

4. Conclusion

- This study underscores the critical role of environmentally friendly catalysts in enhancing oil recovery and upgrading heavy oil. By utilizing recycled industrial metals, we introduced an innovative approach to catalyst preparation that not only enhances process efficiency but also mitigates environmental impacts. Among the catalysts studied, Fe_3O_4 nanocatalysts synthesized via the co-precipitation method and catalysts prepared through chemical and biological leaching methods were extensively characterized and evaluated for their performance.
- A general factorial design of experiments was employed to optimize the catalyst type, concentration, and temperature for maximum effectiveness. Under optimal conditions, the viscosity of the heavy oil was reduced by 10.51 %, and the oil mass decreased by 0.248 % compared to the untreated sample. This viscosity reduction was attributed to the electrostatic stabilization of asphaltene particles by the catalysts.
- The FTIR results of the oil samples showed that the aromatic groups were reduced for all catalysts, which can be a good indicator for upgrading the oil containing these catalysts. Also, the addition of bioleaching solution to the crude oil indicated the promotion of catalyst due to the fact that the aliphatic groups of the oil increased.
- The dynamic analysis of the effect of catalysts on enhancing oil recovery shows that catalysts can be very effective in the rate of oil recovery from reservoirs. The difference in oil recovery factor before and after the upgrading process for the injected solutions of water, nano Fe_3O_4, and the biological catalyst is 9 %, 17 %, and 11 %, respectively.
- To improve the competitiveness of bioleaching, further optimization is needed, focusing on: exploring different microbial strains; adjusting nutrient conditions to enhance microbial activity; and employing two-step bioleaching strategies, which separate metabolite production from the leaching phase. Enhancing bioleaching efficiency is crucial as it reduces the reliance on harsh chemicals, which are often used in chemical leaching, thereby improving the economic and environmental sustainability of the process through lower costs, reduced energy consumption, and minimized waste generation.

CRediT authorship contribution statement

Mohammad Hossein Shabani: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Data curation, Conceptualization. **Parya Torkaman:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Data curation, Conceptualization. **Hamidreza Farshadfar:** Writing – review & editing, Writing – original draft, Validation,

Software, Methodology, Investigation, Conceptualization. **Arezou Jafari:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Funding acquisition, Formal analysis. **Seyyed Mohammad Mousavi:** Writing – review & editing, Supervision, Software, Project administration, Methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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