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DESIGN OF A LABORATORY SETUP TO DETERMINE THE DISPERSION PARAMETER IN A SINGLE-WELL CHEMICAL TRACER TEST

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ABSTRACT

The Single Well Chemical Tracer Test (SWCTT) is a precise in-situ method for estimating residual oil saturation (S_{or}) based on the chromatographic delay between a partitioning tracer (ester) and its hydrolyzed product (alcohol). In this study, laboratory flooding experiments were designed and conducted in a glass-bead packed system at a constant temperature of 149°F to determine the dispersion and kinetic parameters of Ethyl Acetate (EtAc). The experiments were performed under two salinity conditions (5,000 ppm and 50,000 ppm) and simulated using a 1D model in the UTCHEM simulator with a 1 mm cell size. The results demonstrated that salinity significantly influences reaction kinetics and transport properties. Increasing salinity from 5,000 to 50,000 ppm resulted in an increase in the partition coefficient (K_D) from 3 to 5.1 and the hydrolysis rate (K_H) from 0.1 to 0.5/day. Furthermore, the hydrodynamic dispersion coefficient was determined through matching to be 0.003 ft^2/day for the test conducted at a salinity of 5000 ppm and 0.01 ft^2/day for the test conducted at a salinity of 50,000 ppm, resulting in accurate agreement between the simulated and experimental data. The S_{or} values of 0.2414 and 0.2303 obtained from the combined analysis of the model and experimental data showed excellent agreement with the reference value determined by material balance. This consistency serves as robust validation of the laboratory setup and confirms that the developed methodology reliably quantifies both S_{or} and hydrodynamic dispersion. Crucially, these findings establish a solid foundation for upscaling the methodology to pilot tests and reservoir-scale applications.

KEYWORDS

Single well chemical tracer test (SWCTT), Oil Saturation, Hydrodynamic Dispersion Coefficient, Partitioning Coefficient, Chromatographic, UTCHEM

1. INTRODUCTION

In the natural production of oil from reservoirs, a substantial portion of the oil invariably remains trapped within the porous medium of the reservoir rock. This inherent characteristic means that the initial yield of extractable oil from these reservoirs is often relatively low, leaving significant volumes of valuable resources unrecovered. To address this challenge and enhance oil recovery, various techniques have been developed. Among these, waterflooding stands as the most widely adopted and commonly employed method (10). This process involves injecting water into the reservoir to displace and drive the oil towards production wells, thereby augmenting the total oil output. However, even after the waterflooding, a considerable amount of oil persists within the porous rock, referred to as residual oil saturation (S_{or}). This trapped oil is difficult to mobilize due to capillary forces and intricate surface interactions between the oil, water, and reservoir rock. Significant concerns exist regarding the distribution of residual oil after waterflooding, potentially making further oil displacement or Enhanced Oil Recovery (EOR) methods economically unattractive. Therefore, determining the accurate amount of residual oil saturation is one of the most important parameters in the design, potential evaluation, and successful implementation of an EOR method. Indeed, depending on the volume and distribution of this remaining oil, petroleum engineers can judiciously select the most effective EOR method to liberate these trapped hydrocarbons, ultimately maximizing the ultimate oil recovery from the reservoir (1, 9).

Currently, a variety of techniques are available to ascertain this key parameter. However, core analysis and well logging stand out as the two most prevalent and well-established methods for determining S_{or}. While both core analysis and well logging provide invaluable data, each technique possesses inherent limitations that must be carefully considered. A primary shared drawback of both approaches is that the values they yield represent averages over very small reservoir volumes. Specifically, core analysis examines only a small sample of reservoir rock extracted from a particular depth, while well logs gather information from the relatively confined vicinity around the wellbore. This volumetric limitation implies that the obtained results do not necessarily reflect the overall characteristics of the entire reservoir and may fail to accurately capture the complex heterogeneities and variations present on a larger reservoir scale. Consequently, this inherent uncertainty in S_{or} measurement can impact the accuracy of reservoir performance predictions and the optimal selection of EOR methods, thereby underscoring the critical need for the development of more precise and comprehensive measurement techniques (14).

The Single Well Chemical Tracer Test (SWCTT) stands as a highly reliable and precise in-situ technique for estimating S_{or} within a range of 20 to 30 meters from the wellbore. This method overcomes many of the limitations inherent in other techniques, such as core analysis and well logging. This superior accuracy and reliability position SWCTT as a valuable tool in reservoir engineering (7, 8). The historical application of SWCTT dates to 1968, when Deans and his colleagues at Esso Production Research Company successfully launched the first field application of these tests in the East Texas Field. This pioneering effort paved the way for the subsequent development and refinement of the technique (3, 4). Saturation measurement with tracers relies on the principle of chromatographic retardation using two types of tracers. One of these tracers can dissolve in both water and oil, while the other tracer can only dissolve in water. Most of the reported SWCTT operations use ethyl acetate (EtAc) as the primary tracer, normal propyl alcohol (NPA) and isopropyl alcohol (IPA) as the cover tracer and mass balance tracers (5). The primary tracer bank consists of a reactive ester (partitioning tracer) that is injected into the formation, followed by a push volume (bank of tracer-free water) to place the ester at a certain distance from the wellbore. The well is temporarily shut down for a specific period to allow the ester to hydrolyze and form the secondary tracer (alcohol). When production resumes, both the primary and secondary tracers move toward the well. This method exploits the time lag of back-produced ester versus hydrolyzed alcohol. The time delay is due to chromatographic separation between the secondary tracer (alcohol) and partitioning tracer (ester) and is related to the saturation of a

stagnant phase (oil) and the tracer's partition coefficients (K_d) (11). By meticulously monitoring the effluent concentrations of both the primary and secondary tracers, the residual oil saturation can be accurately determined from this time lag between their respective concentration peaks. This characteristic makes SWCTT a powerful instrument for gaining a deeper understanding of oil saturation conditions within reservoirs.

In this study, to accurately investigate the performance of the SWCTT, a laboratory flooding system was first designed and set up to provide reliable experimental data under controlled conditions. Subsequently, a comprehensive numerical model was developed using the UTCHEM simulator to simulate the entire SWCTT process, including tracer injection, the hydrolysis reaction in the porous medium, and the production of reaction products. The primary objective of this research was to identify and evaluate the parameters influencing the hydrolysis reaction kinetics of the Ethyl Acetate (EtAc) tracer and to determine optimal values for the partition coefficient (K_D), hydrolysis rate (K_H), and dispersion coefficient through matching with laboratory data. The model developed in this research can serve as an efficient tool for predicting the behavior of reactive tracers and optimizing the design of SWCTT operations in future studies.

2. MATERIALS AND METHODS

2.1. Experimental materials and reagents

All chemicals were of analytical grade (ACS) and used as received without further purification. Ethyl acetate ($C_4H_8O_2$), normal propyl alcohol (C_3H_8O), isopropyl alcohol (C_3H_8O), Ethanol (C_2H_5OH), polyethylene glycol (PEG) and sodium chloride (NaCl) were commercially obtained[a] with purities of >99%. Synthetic brines were prepared by dissolving inorganic salt (NaCl) in deionized (DI) water. Crude oil (dehydrated, without mechanical impurities, 29 API) used in this research was obtained from an active oil field in southern Iran.

2.2. Tracer detection

To determine the concentration of tracers by gas chromatography, standard solutions were prepared with different known concentrations of primary and secondary tracers and calibration curves were plotted. The prepared standard solutions were transferred into vials with disposable fluoroplastic screw caps. All measurements were performed on a gas chromatograph[b] equipped with a flame ionization detector (FID) and an automatic headspace sampler (HS-GC/FID). Separation was achieved using a DB-ALC1 column[c] (30 m × 0.53 mm × 3 µm).

2.3. Determination of hydrolysis rate and partitioning coefficient

To determine the hydrolysis rate and partition coefficient of ethyl acetate as a chemical tracer, a known and precisely measured amount of ethyl acetate was initially dissolved in synthetic brine. For hydrolysis rate determination, the tracer-containing brine was maintained under reservoir conditions. Samples were collected at 1-hour intervals and analyzed using gas chromatography. The hydrolysis rate was quantified based on the decrease in ethyl acetate concentration in the aqueous phase and the simultaneous formation of the secondary tracer (ethanol).

For partition coefficient determination, the tracer-containing brine was added to crude oil at predefined volumes. The aqueous and oleic phases were thoroughly mixed using a high-speed mechanical stirrer to ensure complete homogenization. After mixing, the system was kept under

[a] *Sigma Aldrich Co. (Merck, Germany): https://www.sigmaaldrich.com*
[b] *Agilent 6890N gas chromatograph: https://www.agilent.com/cs/library/specifications/public/5989-3290EN.pdf?srsltid=AfmBOoq9o43VJAH8_f1uQsPr6RNx17yRmb56-nBPY-sTesmaG4XvwlHx*
[c] *Agilent J&W DB-ALC1 column: https://www.agilent.com/en/product/gc-columns/application-specific-gc-columns/db-alc1-db-alc2-columns?srsltid=AfmBOorTCU6hHFRI7nCc4JKRENUxQwMy7Yl64MT7jPoY07DGvKH_Spos*

reservoir conditions until thermodynamic equilibrium between oil and water phases was attained. Samples were collected at 2-hour intervals and analyzed using gas chromatography.

2.4. Flooding experiments

A laboratory flooding system was designed and set up to determine the dispersion and adsorption parameters relevant to single well chemical tracer tests (**Fig. 1**). The system consisted of a syringe pump[d] equipped with a 100 mL syringe (inner diameter of 32.57 mm) to ensure constant and accurate injection rates. Temperature control was achieved using a circulating water bath[e] with temperature-regulated water continuously flowing through a thermal jacket surrounding the packed column to maintain reservoir-representative conditions. The column had a length of 150 cm and an inner diameter of 0.9 cm, and was uniformly packed with glass beads of mesh size 25 to form a homogeneous porous medium. The resulting porous medium exhibited an absolute water permeability of 42 ± 0.5 D, which was determined using Darcy's law under single-phase flow conditions. Porosity of 37.2% was determined by injecting water into the packed column and quantifying the pore volume (PV). Before the injection of primary bank, the porous medium was prepared to establish a stable residual oil saturation (S_{or}). This detailed saturation procedure began by injecting 3 PVs of crude oil at a constant flow rate (0.6 mL/min) to achieve full saturation. Subsequently, 25 PVs of brine (with a total salinity of 5 mg.L^{-1}) were then injected into the system at the same flow rate. This water-flooding process was continued until oil ceased to be observed in the effluent, confirming the establishment of the stable S_{or}. The S_{or} was quantitatively

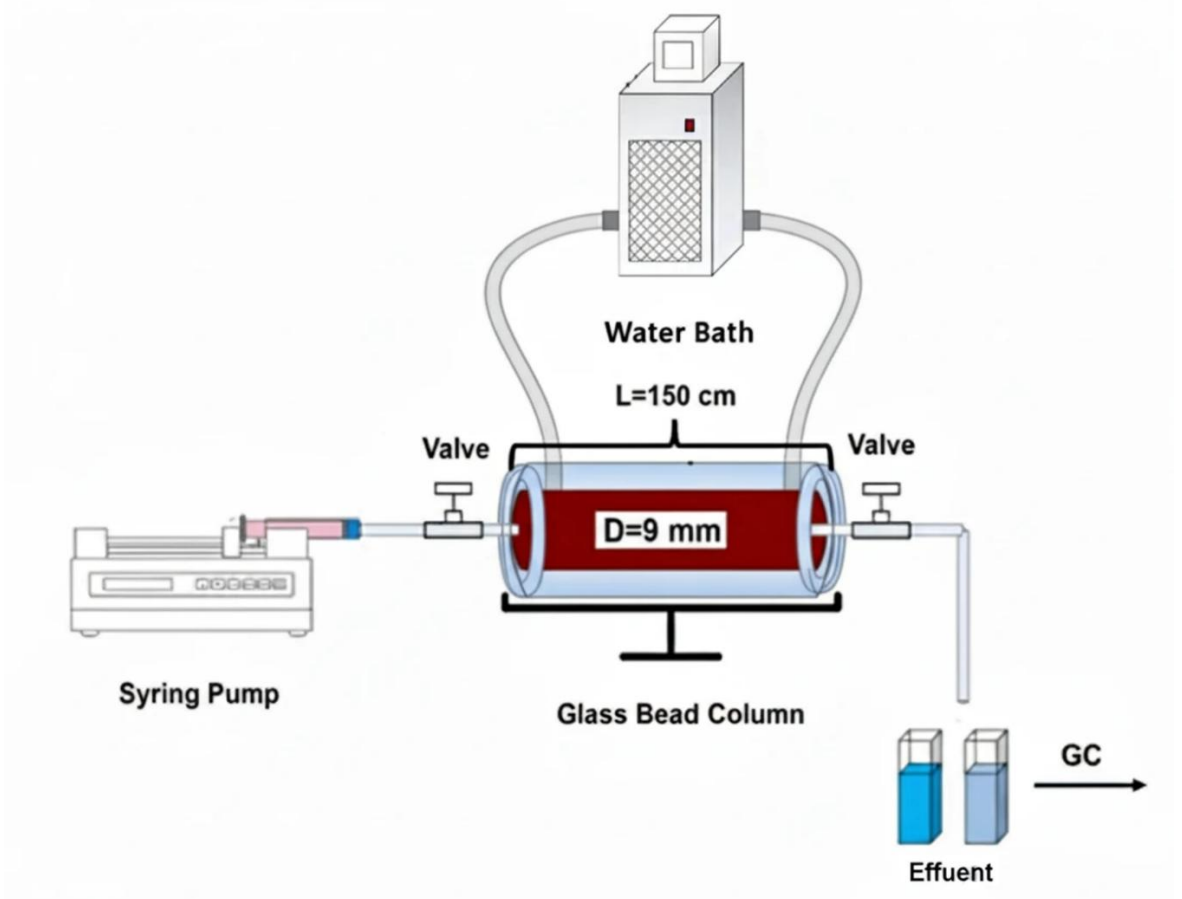


Figure 1: Schematic of the laboratory flooding system.

[d] *FNM SP102: https://fnm.ir/products/Syringe_Pump*
[e] *Dorsatech DCB-02: https://dorsatech.ir/en/*

determined as 25.35% using a material balance approach, based on the difference between the total injected oil volume and the cumulative oil volume produced in the effluent.

The experiment was performed at a constant temperature of 149°F. Primary tracer bank, consisting of EtAc, IPA and NPA, was injected into the porous medium at their respective optimal concentrations of 1% v/v, 0.25% v/v, and 0.5% v/v, under a constant flow rate (0.6 mL/min). Following the primary tracer bank, push bank containing the same concentration of the IPA tracer (0.25% v/v) was injected at the same flow rate to displace the tracer bank and establish favorable conditions for subsequent shut-in. After a shut-in period that allowed for the generation of the secondary tracer through ethyl acetate hydrolysis, effluent samples were collected at predetermined production intervals at a constant production rate of 0.4 mL/min, with each sample having a volume of 1.5 mL. The concentrations of the tracers in the collected effluent samples were determined using a gas chromatograph. Based on the analytical results, tracer concentration profiles were constructed as a function of cumulative liquid production for subsequent interpretation.

To reduce analytical costs while maintaining measurement accuracy, an alternative sample preparation method was developed and implemented. To effectively mitigate risks and prevent potential damage to the chromatographic column and detector, 1.5 cc of the aqueous solution was diluted in 23.5 cc of a solvent. This controlled dilution ensured that the water content in the final injected sample was reliably reduced to less than 5.5% w/w, thereby preventing interference with the chromatographic separation and detection processes, and safeguarding the analytical equipment. The measurements were performed on a gas chromatograph[f] with a flame ionization detector (FID) and Supelco BP5 column (30 m × 0.22 mm × 0.25 µm). Helium of 99.999% purity served as the carrier gas at a flow rate of 3 mL/min. The injection volume was 1 µL and injector and detector temperature were set at 250°C. The oven temperature was initially held at 40°C for 1 min, then increased to 48°C at a rate of 0.5°C/min and held for 1 min. Finally, the temperature was ramped to 250°C at 150 °C/min and maintained for 8 min.

2.5. SWTT design

UTCHEM software[g] was used for modeling because it is a three-dimensional, multiphase, multicomponent flooding simulator with the capability to model reactive partitioning tracers, as well as surfactant, polymer, and alkaline injection (2, 13). For simulation, a 1-D homogeneous cartesian model with a horizontal cell size of 1 mm was used, as this cell size yielded the best match between modeled and measured data. Previous studies have shown that cell size does not affect the position of the tracer concentration peak on the produced volume axis, but it does influence the magnitude of the peak concentration (6).

For simplification, the system was simulated at a constant temperature of 149°F. Selecting an appropriate reactive primary tracer and determining parameters such as its partition coefficients (K_d) and hydrolysis reaction rate (K_H) are of particular importance. Reservoir temperature is considered a key factor in identifying the optimal primary tracer. In this study, the usability of various chemical compounds was investigated. Two common reactive ester compounds in the industry are normal propyl formate (NPF) and ethyl acetate (EtAc). Normal propyl formate is typically recommended for low-temperature reservoirs, while EtAc is advised for high-temperature reservoirs (12). Given the high temperature of the system (149°F), EtAc was selected as the primary tracer due to its suitable reaction kinetics at this temperature. To achieve more accurate modeling, the kinetic parameters of the EtAc reaction, such as its hydrolysis rate, hydrodynamic dispersion coefficient (D_H), and partition coefficient, must be determined. These parameters can be calculated through separate experiments or by utilizing empirical methods

[f] *Shimadzu 2010: https://www.shimadzu.com*

[g] *https://csee.engr.utexas.edu/research/industrial-affiliate-programs/chemical-enhanced-oil-recovery/ut-chem-simulator*

available in the literature. Temperature and salinity significantly influence both the partition coefficient and hydrolysis rate; an increase in either of these parameters leads to a corresponding rise in both values. These parameters were used for accurate simulation of hydrolysis reaction kinetics in the UTCHEM model. To determine the optimal operational parameters, two distinct experiments were designed. These experiments were conducted at salinities of 5,000 ppm and 50,000 ppm, while maintaining a constant temperature of 149°F. To accurately assess the tracer's behavior within the porous medium, its partition coefficient and hydrolysis rate were determined under both designated test conditions.

Since the experiment was conducted in a laboratory flooding system, scale effects were not significant. However, in field studies, scale effects must be considered. Temperature, pH, salinity, tracer diffusion radius in the rock matrix, and reaction time are among the important factors that can influence tracer reaction kinetics. Other assumptions considered in this modeling include:

a) the injected fluid was assumed to be single-phase aqueous and incompressible;
b) thermal effects were neglected, and the system was assumed to be isothermal;
c) no side reactions or gas production were considered in the system; and
d) petrophysical properties were assumed to be homogeneous and independent of pressure.

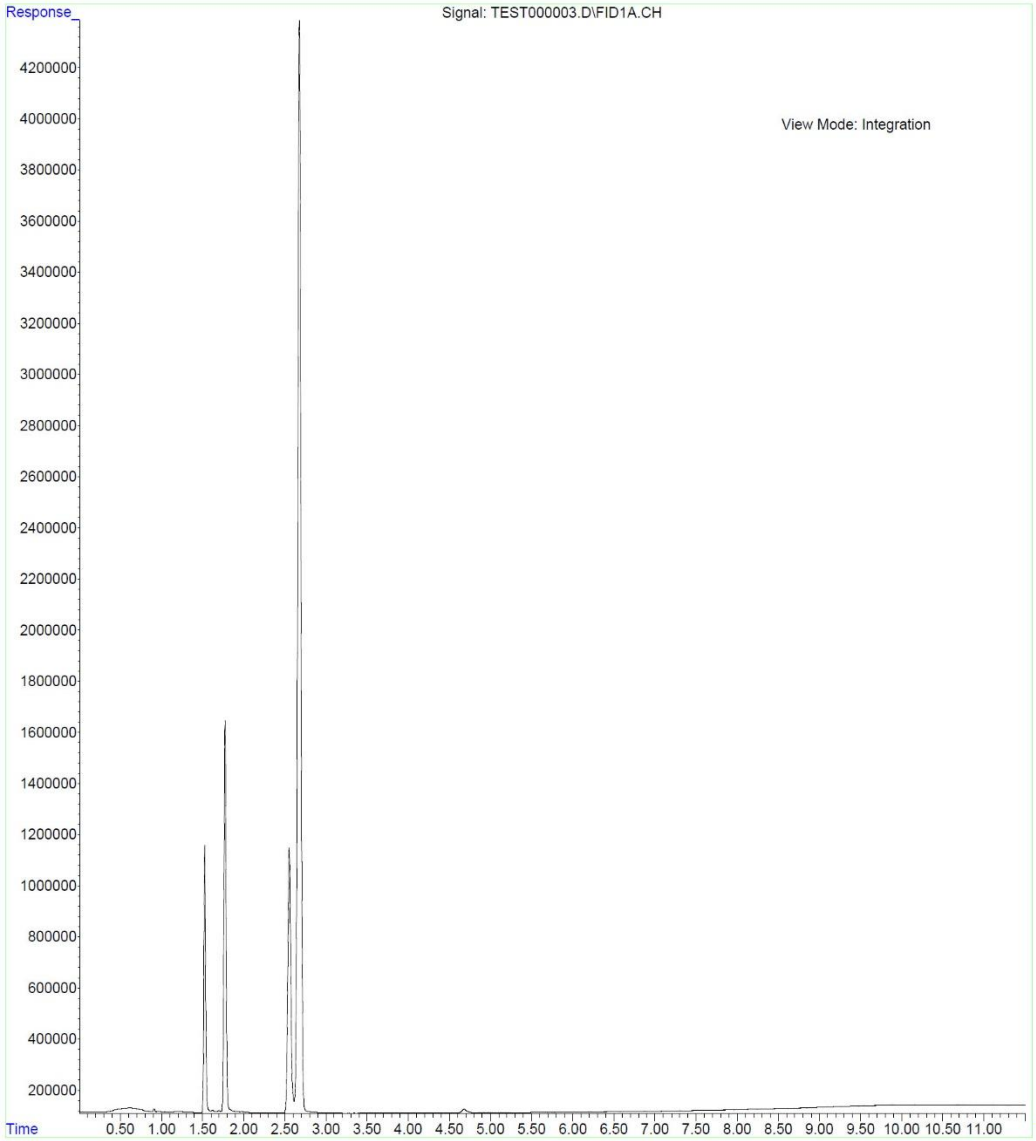
These assumptions are typically made to simplify the model and reduce computational complexity. In future studies, the model can be brought closer to reality by considering more complex effects. After defining the necessary inputs, the UTCHEM model was run to simulate the injection, hydrolysis reaction, and production process in the SWCTT experiment. The simulation results were then used in comparison with the measured laboratory data.

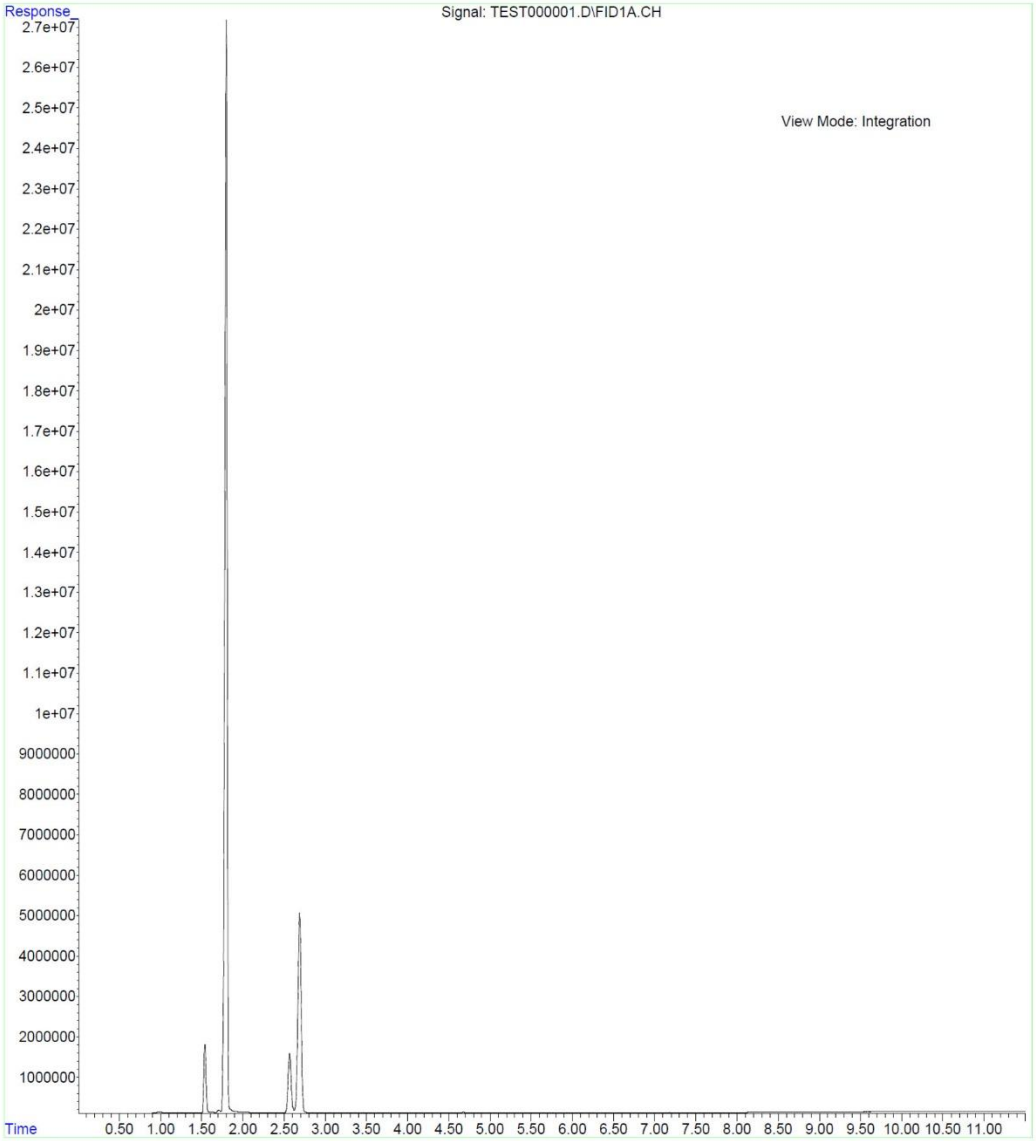
3. RESULT AND DISCUSSION

3.1. Chromatographic Separation and Calibration

The reliability of SWCTT data hinges on the accurate detection of tracer concentrations. The Gas Chromatography (GC) method successfully separated the primary tracer (Ethyl Acetate), the secondary tracer (Ethanol), and the mass balance and cover tracers (IPA and NPA). As illustrated in the chromatograms obtained from the headspace sampler, distinct peaks were recorded for each component with stable retention times (**Fig. 2**). This separation confirms that there was no co-elution or interference between the tracers, which is critical for accurate concentration profiling. To quantify these concentrations, calibration curves were plotted for all four tracers. The results exhibited excellent linearity across the experimental concentration ranges, validating the analytical method's precision for this study (**Fig. 3**).

Figure 2: Typical chromatogram obtained for a standard sample. The elution order of the peaks (in order of increasing retention time) is: Ethanol (EtOH), Isopropyl Alcohol (IPA), n-Propyl Alcohol (NPA), and Ethyl Acetate (EtAc). Figure can be downloaded here.





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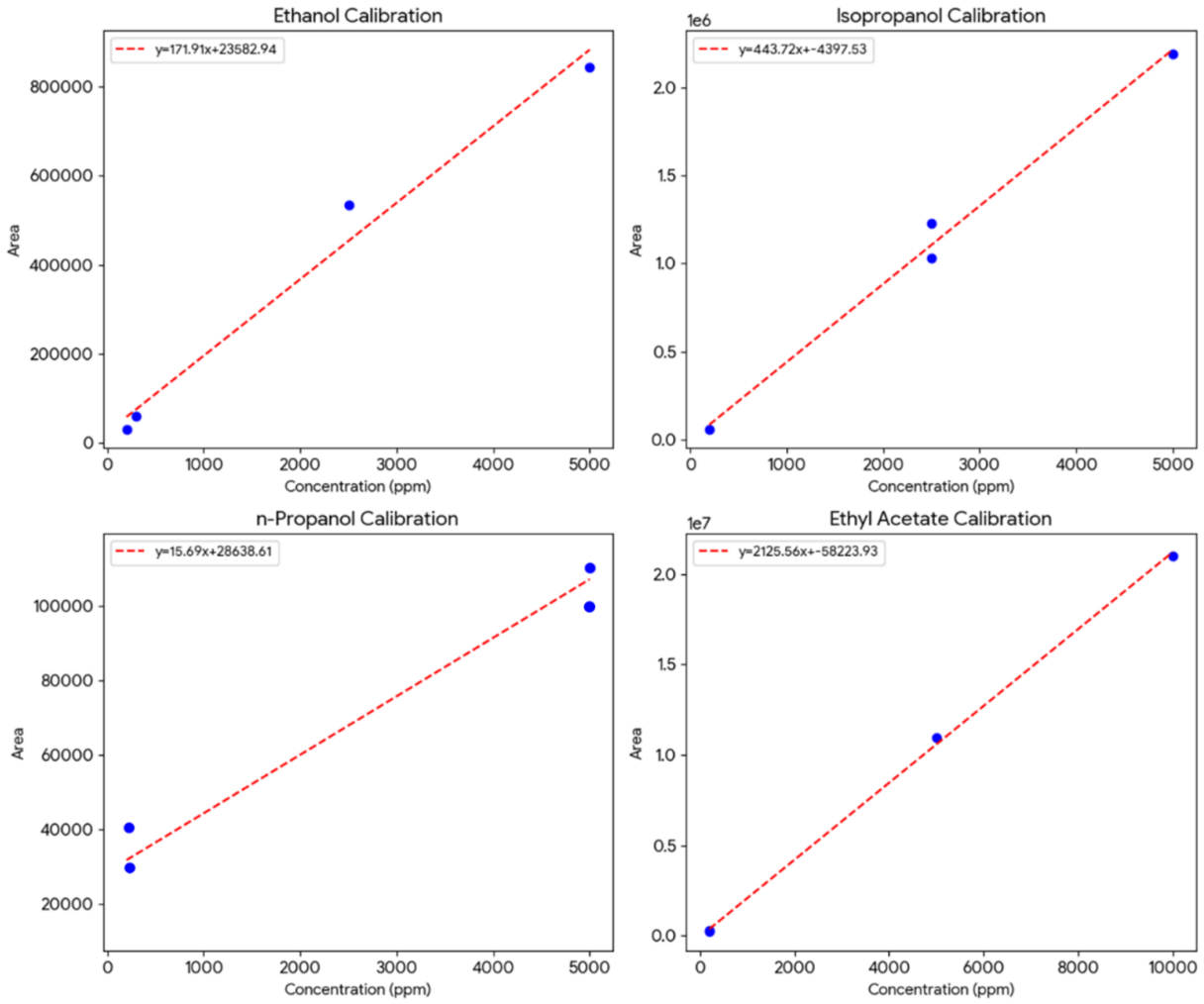

Figure 3: Calibration curves exhibiting excellent linearity for all four tracers across the experimental concentration ranges.

3.2. Determination of kinetic parameters

The accuracy of the SWCTT interpretation relies heavily on the precise determination of the reaction kinetics under representative reservoir conditions. The two critical parameters governing the process are the partition coefficient (K_D), which dictates the chromatographic separation, and the hydrolysis rate constant (K_H), which determines the generation of the secondary tracer. Laboratory experiments must examine the equilibrium criteria for tracers in both aqueous and oleic phases to obtain reliable K_D values for field interpretation. In this study, kinetic parameters for the primary tracer, EtAc, were determined under two distinct salinity conditions (5,000 ppm and 50,000 ppm) while maintaining a constant reservoir temperature of 149°F. This temperature range was selected as EtAc exhibits suitable reaction rates for these thermal and salinity conditions. The experimental results revealed a direct correlation between salinity and kinetic parameters. At the lower salinity of 5,000 ppm, K_D was determined to be 3, with a K_H of 0.1/day. Conversely, at 50,000 ppm, a significant increase in both parameters was observed, yielding a K_D of 5.1 and a K_H of 0.5/day.

3.3. Determination of S_{or}

The key experimental specifications, optimized injection and production parameters, and the determined hydrolysis reaction kinetics of the ethyl acetate tracer for the first and second tests are summarized in **Table 1** and **Table 2**, respectively.

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Table 1: The measured laboratory specifications for the first and second tests

Test	Salinity of Brine (ppm)	K_D	$K_H(day^{-1})$	Porosity	Permeability (D)	S_{or}
1	5,000	3	0.1	0.372	42	0.2535
2	50,000	5.1	0.5	0.4034	51	0.2338

Table 2: Optimized parameters in the tracer injection and production process during the Single Well Chemical Tracer Test (SWCTT)

Test	Injection rate (ml/day)	Production rate (ml/day)	Primary bank (PV)	Push bank (PV)	Injection period (hr, PV)	Shut-in time (hr)	Production period (hr, PV)
1	864	576	0.116	0.684	0.86, 0.8	84	2.4, 1.5
2	864	576	0.116	0.684	0.86, 0.8	21	2.4, 1.5

Figure 4 presents the experimental and simulated concentration profiles for EtAc, NPA, IPA, and EtOH. These profiles are plotted as a function of the produced pore volume (PV) for both the first and second experimental tests. As anticipated, the observed inverse relationship between the decreasing EtAc concentration and the concurrent increase in EtOH concentration (evident in **Fig. 4**) confirms the progression of the hydrolysis reaction. In the second test, the higher salinity enhanced both the partition coefficient and the hydrolysis rate of the EtAc tracer. Consequently, a decrease in the concentration of the primary tracer was observed, accompanied by a corresponding increase in the concentration of the secondary tracer.

Following the application of dead volume corrections, specifically 0.113 PV (4.02 mL) for the first test and 0.10 PV (3.85 mL) for the second test, a good agreement was observed between the experimental data and the simulated results for both tests, as depicted in **Figure 5**. It should be noted that the identified dead volumes mainly originated from fluid residence within the tubing and delays caused by sampling errors. Additionally, the adsorption coefficient was assumed to be negligible in the simulations.

After correcting for the dead volume, the experimental data from the first test exhibited excellent matching with the corresponding simulation results. For this test, the dispersion coefficient was estimated to be 0.003 ft^2/day, which provided an excellent match between the modeled and measured effluent concentration profiles. In the second test, although an overall reasonable agreement was initially observed, the experimental tracer profiles displayed noticeably flatter and broader peaks with lower maximum concentrations compared to the initial simulation results. This behavior is attributed to the increase in salinity in the second test, which enhanced hydrodynamic dispersion. As a result, a higher dispersion coefficient of 0.01 ft^2/day was required to properly capture the observed spreading of the tracer plume. Incorporation of this corrected dispersion coefficient in the refined simulation led to an excellent match between the simulated and experimental results.

The value for S_{or} was determined from the difference in retention times between the peaks of the secondary tracer and the partitioning tracer. This analysis yielded S_{or} values of 0.2414 ± 0.0016 for the first test and 0.2303 ± 0.0046 for the second test. These determined values are in excellent agreement with the reference S_{or} value previously determined for the glass pack under laboratory conditions, confirming the reliability of the experimental approach.

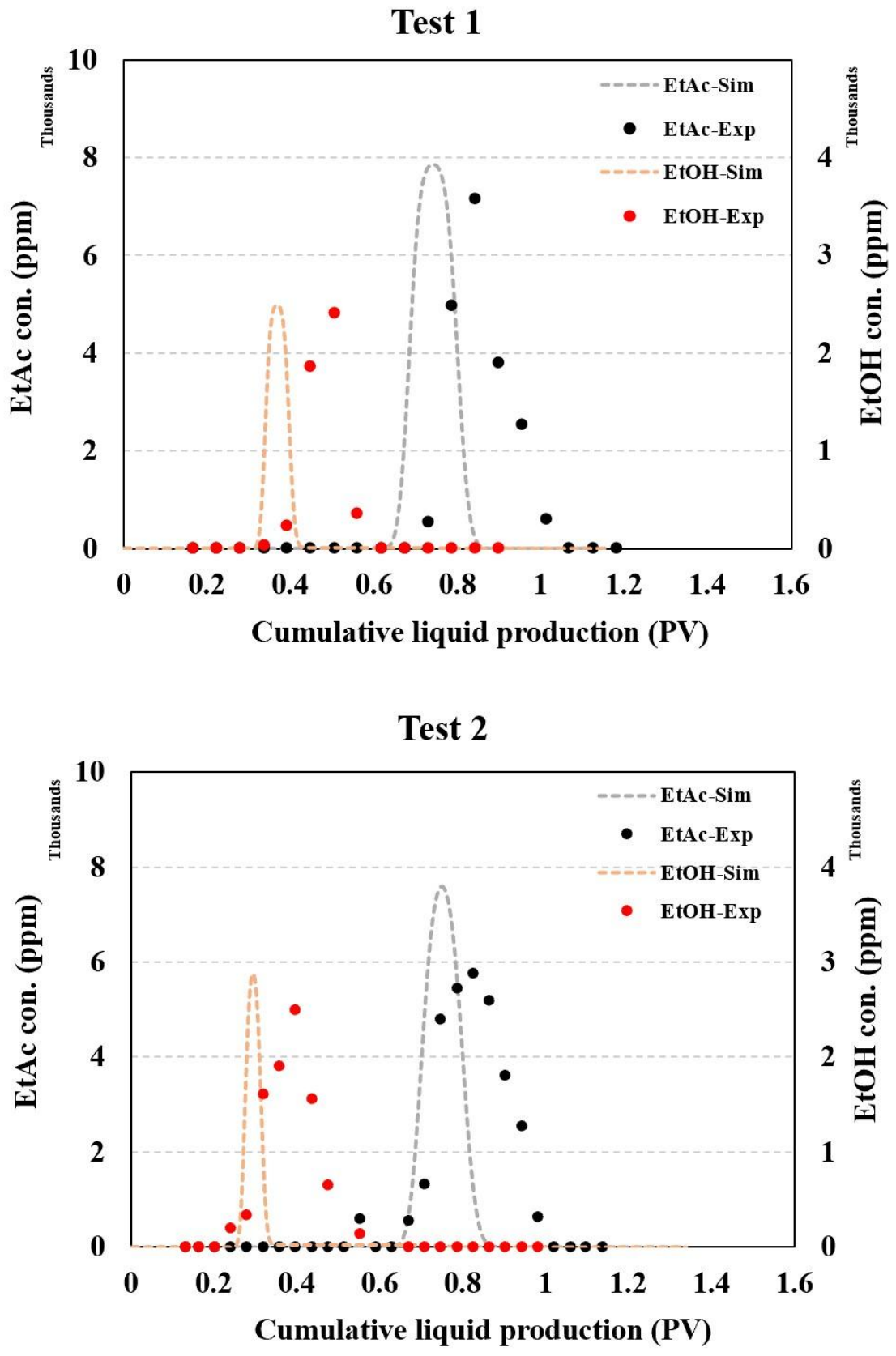

Figure 4: Initial laboratory and simulation results of Tests 1 and 2.

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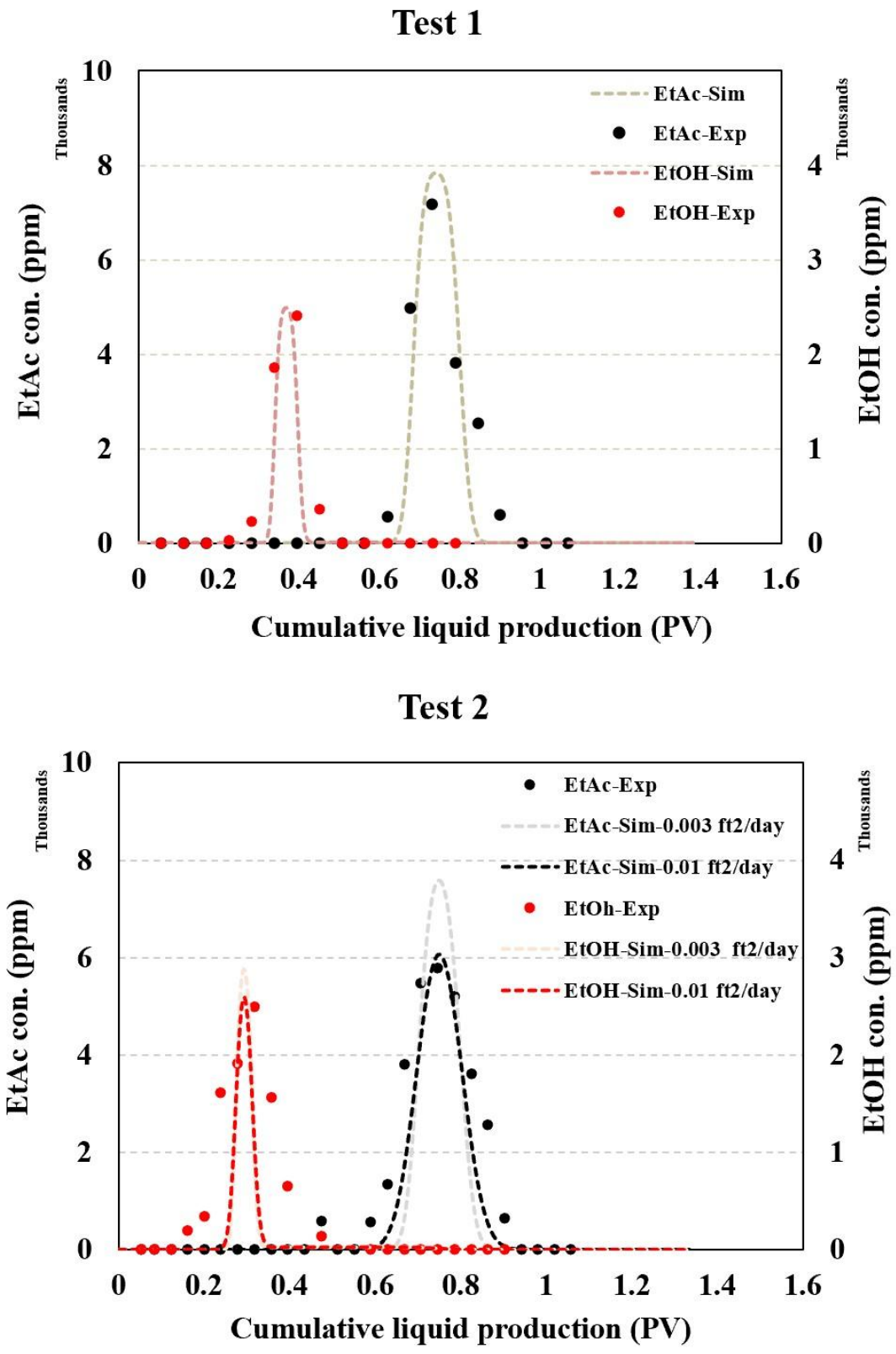

Figure 5: Match between simulation results and laboratory data.

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4. SUMMARY AND CONCLUSIONS

This study presents an integrated experimental and numerical investigation of SWCTT using a glass bead pack column and the UTCHEM reservoir simulator. The developed laboratory setup and modeling methodology successfully reproduced the full SWCTT process, including tracer injection, chromatographic separation, in-situ hydrolysis of the primary tracer (ethyl acetate, EtAc), and secondary tracer production. The close agreement between experimental data and simulation results confirms the validity of the experimental design and the robustness of the numerical model.

The hydrodynamic dispersion coefficient was determined through history matching to be 0.003 ft^2/day for the low-salinity test (5,000 ppm) and 0.01 ft^2/day for the high-salinity test (50,000 ppm), accurately capturing the observed differences in tracer peak spreading. Adsorption effects were assumed to be negligible under the experimental conditions. Residual oil saturation (S_{or}) was determined from the difference in retention times between the partitioning and secondary tracers. The S_{or} values obtained from the combined analysis of experimental data and simulation modeling were 0.2414 ± 0.0016 and 0.2303 ± 0.0046, which showed excellent agreement with the reference values determined by material balance. These results demonstrate that the laboratory setup was properly validated and that the developed methodology enables reliable quantification of both residual oil saturation and hydrodynamic dispersion.

The close agreement between the experimentally determined S_{or} values and the reference data serves as robust validation of the laboratory setup. Moreover, the consistency between experimental observations and simulation results confirms that the developed methodology reliably quantifies both S_{or} and the hydrodynamic dispersion coefficient. Crucially, these findings provide a solid foundation for upscaling the methodology and inferred parameters to pilot tests and reservoir-scale applications, thereby extending the approach's applicability beyond laboratory conditions. Additionally, the results demonstrate that the developed model accurately captures both the transport dynamics and hydrolysis reaction kinetics of the EtAc tracer. This underscores the critical importance of determining precise kinetic parameters and employing rigorous modeling for the accurate interpretation of SWCTT data.

Additionally, to fully validate the results and ensure their reliability for field pilot applications, it is highly recommended to utilize reservoir rock (such as sandstone or carbonate) in future experiments. Furthermore, the adsorption parameter should be explicitly included in the analysis to account for rock-fluid interactions, thereby providing a more accurate basis for upscaling to field conditions.

Although ethyl acetate (EtAc) is a commonly employed primary tracer, its application is constrained by significant limitations, including flammability, high volatility, considerable cost, and inherent safety concerns. Consequently, the exploration and adoption of novel alternative tracers possessing superior properties are strongly advocated for future investigations. These advanced tracers present distinct advantages over conventional options like EtAc. Key among these are the substantially reduced injection volumes and concentrations required, coupled with their detectability at extremely low levels, often in the parts-per-billion (ppb) range. Such attributes lead to marked reductions in tracer-associated expenditures and notable improvements in operational safety. Therefore, to improve the accuracy, safety, and cost-effectiveness of SWCTT, further research is proposed to identify, evaluate, and optimize the use of these innovative tracers. Concurrently, developing simulation models specifically for these new tracers will be crucial in better interpreting the results of future SWCTT experiments.

STATEMENTS AND DECLARATIONS

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Author Contributions
M. Ramezani: Conceptualization, Methodology, Investigation, Simulation, Formal analysis, Writing – Review & Editing. **R. Miri:** Supervision**,** Project administration, Writing – Review & Editing**.**

Conflicts of Interest
There are no conflicts of interest to declare.

Data, Code & Protocol Availability
The data can be provided upon request.

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