



Thermal stability and interactions with sedimentary rocks under typical reservoir conditions of selected pyridines investigated as phase partitioning tracers

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ABSTRACT

Pyridine, 2-hydroxy-6-methylpyridine, 3-hydroxypyridine, and 4-methoxypyridine are evaluated as potential phase-partitioning oil field tracers. Their stability is tested in a brine for 12 weeks at temperatures between 25 °C–150 °C, and at initial pH values of 5.5; 7.1; 8.0. Interactions with kaolinite clay, Berea sandstone and limestone are also evaluated. The main results are as follows: pyridine is stable up to 12 weeks at 150 °C, and not influenced by the rock substrates or pH. 2-hydroxy-6-methylpyridine becomes unstable at $T \geq 50$ °C, is not affected by the rock substrates, and exhibits slower degradation kinetics at higher pH values. 3-hydroxypyridine is unstable at $T \geq 75$ °C, sensitive to the presence of kaolinite in a combined effect with pH, and exhibits slower degradation kinetics at higher pH. 4-methoxypyridine degrades at $T \geq 75$ °C, is characterised by a strong interaction with kaolinite, and is insensitive to pH.

The degradation of 4-methoxypyridine in the absence of kaolinite clay follows pseudo first-order kinetics. This compound could indicate the temperature in the swept volumes, and in conjunction with a fully conservative tracer indicate the presence of clays. Pyridine exhibits the required stability and lack of interaction with rock materials to be used as PITT tracer in oil reservoirs. However, it is present in oils and its concentration levels in production waters should be evaluated prior to its use.

1. Introduction

Knowledge of the residual oil saturation (S_{OR}) in the swept volumes of oil reservoirs can be used both to improve reservoir management, and in the design and evaluation of EOR/IOR projects [1,2]. As the number of mature fields increases [3], such projects become more important to ensure the production of hydrocarbons to satisfy global demand. A partitioning inter-well tracer test (PITT) is the only dynamic tool to measure S_{OR} in the inter-well region of waterflooded reservoirs. A PITT is based on the use of passive and oil/water partitioning tracers [4]. It was introduced to the oil industry in 1971 by Cooke [5], however never routinely used. The poor knowledge about the behaviour and geochemical interactions of the compounds used as PITT tracers lead to many unsuccessful tests in the past [4,6]. In recent years, this type of tracer test has received increasing attention due to the importance of the information it provides [7,8]. Additionally, a small number of compounds has been developed for use as PITT tracers [6,9]; additional PITT tracers would be useful. The study of tracer compounds prior to their use on the field

is crucial, not only to ensure the accuracy of the tests, but also to evaluate the possibility of using tracers to retrieve other type of relevant information [10–12].

In this document we present the stability assessment performed on 4 pyridines that are PITT tracer candidates. The influence of temperature, pH, time, salinity, and different rock materials, on the stability and behaviour of pyridine, 4-methoxypyridine, 3-hydroxypyridine, and 2-Hydroxy-6-methylpyridine was investigated.

1.1. Pyridines

Pyridine has the chemical formula C_5H_5N and a structure similar to benzene. It is a weakly basic, transparent “yellowish”, flammable, and water-soluble heterocyclic aromatic compound, with a distinct highly unpleasant smell. Such properties are also observed in many of the different substituted pyridines. Many compounds within this family of chemicals show interesting properties for use as PITT tracers. Relevant properties of the compounds tested here can be found in Table 1.

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Table 1

Melting point, boiling point, pKa, and octanol/water partitioning coefficient of the pyridines selected as partitioning tracer candidates.

Compound	Melting point (°C)	Boiling point (°C)	K _{OW}	pKa
Pyridine	-42	115	4,47	5,25*
4-Methoxypyridine	4	191	5,22	6,58*
3-Hydroxypyridine	126	329	4,36	4,79*/8,75
2-Hydroxy-6-methylpyridine	159	275	2,18	4,94*/9,27

*Value for the mono-protonated pyridine.

Pyridine and polycyclic pyridine derivatives have been reported in crude oils and are commonly classified as part of their alkaline fraction [13–15]. They are responsible for 20%–26% of the total nitrogen content in all crude oils [13,14] which is usually $\leq 1\%$ (wt), and $< 0.25\%$ (wt) in about 90% of them [13]. The other pyridine derivatives investigated under the scope of this work have never, to the best of our knowledge, been reported as constituents of any type of oil reservoir fluid. The chemical industry uses pyridine as raw-material and intermediate on a large scale. It is the precursor to some pesticides, such as paraquat and diquat [16], industrially used as solvent, in the production of dyes, explosives, pharmaceuticals, etc. [17,18], and is used as a ligand (or base unit for ligands) in coordination chemistry [19]. 4-Methoxypyridine is mostly used as a ligand in coordination chemistry [20,21], and in the synthesis of pharmaceutical compounds [22]. 3-Hydroxypyridine has been considered as a precursor for the production of nitrogen-rich polymers for CO₂ adsorption [23], used as additive to improve optical properties of hydrophilic ophthalmic lenses [24], and also as precursor for pharmaceutical active principles [25]. 2-Hydroxy-6-methylpyridine has no significant industrial use and we only found it reported as a ligand in the preparation of metal-lanthanide complexes or special metal complexes [26,27]. The widespread use of pyridines means that most of them are available at lower cost than the compounds presently used as PITT tracers.

Pyridines are probably one of the least obvious choices for use as PITT tracer candidates because of two main issues: i) pyridine itself has been identified as a minor constituent of crude oils; ii) pyridine and its derivatives are weak bases, and therefore pH sensitive compounds when in aqueous solution. However, the PITT is primarily designed for mature oilfields where waterflooding has been performed for long periods of time (typically years). In such fields, it can be expected that a chemical equilibrium (or quasi-equilibrium) is established between the hydrocarbons and the circulating aqueous phase, which will keep the dissolution of pyridines in the water at a low concentration. Such conditions will not affect the use of pyridines as PITT tracers, as the background “noise” will not compromise detection of the pyridine injected as tracer. A recent study found no evidence of the presence of pyridine in production waters from eight oilfields on the Norwegian continental shelf [28]. The use of deuterated forms of pyridine, could also be used to distinguish the PITT tracer from natural pyridine. The pH sensitivity of this family of compounds could indicate pH conditions inside the porous medium. The partition coefficient (K) of pyridines varies as function of pH.

2. Materials and methods

2.1. Materials

Pyridine ($\geq 99\%$), 4-methoxypyridine ($\geq 99\%$), 3-hydroxypyridine ($\geq 98\%$), 2-hydroxy-6-methylpyridine ($\geq 97\%$), kaolinite (natural aluminum silicate 125 μm –250 μm with linear formula $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) and limestone powder type “BCR-116” (95.7% CaCO_3 + 4.3% MgCO_3) were purchased from Sigma–Aldrich (Sigma–Aldrich Norway AS, 0252 Oslo). Berea sandstone powder [93.13% silica (SiO_2), 3.86% alumina (Al_2O_3), 0.11% ferric oxide (Fe_2O_3), 0.54% ferrous oxide (FeO), 0.25% magnesium oxide (MgO), and 0.10%

calcium oxide (CaO)] (125 μm –250 μm) was obtained by crushing and sieving sandstone cores purchased from Berea Sandstone™ Petroleum Cores (Ohio, USA).

2.2. Experimental procedure

1 L of 10 mg L⁻¹ solution of the selected pyridines was prepared in a synthetic brine. The brine was constituted of 36.855 g L⁻¹ of NaCl, 0.629 g L⁻¹ of KCl, 3.814 g L⁻¹ of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 2.550 g L⁻¹ of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 0.088 g L⁻¹ of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.437 g L⁻¹ of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$, and 0.046 g L⁻¹ of Na_2SO_4 . The pH of this solution was 5.5. Two other solutions to test the compounds at pH 7.1 and 8.0 were prepared using of NaHCO_3 to adjust the pH values.

Oxygen was removed from the solutions through sonication and sparging with 5 mL/min of argon for 20 min. The solutions were kept under constant sparging of argon during the whole time of sample preparation. 4 sets of duxess glass test tubes were prepared: 1 set containing no rock substrate, 1 set containing 600 mg of kaolinite, 1 set containing 600 mg of Berea sandstone, and 1 set containing 600 mg of limestone. 2 mL of the solution of the tracer candidates was transferred to the test tubes. Vacuum was applied to the headspace of the test tubes during their heat-sealing. The experiments were performed in triplicate. The test tubes were then incubated during 12 weeks at temperatures up to 150 °C in thermal cabinets. The thermal cabinets had a rotation mechanism which ensured smooth agitation and homogeneity of the bulk during the experimental period. The concentration of the tested pyridines was monitored after 1, 3, 6, and 12 weeks of incubation. The quantification of the compounds was done by UPLC-UV with detection at 222 and 254 nm, by injecting 10 μL of the test samples filtered through a 0.45 μm PTFE filter into the chromatographic system. A “Agilent Technologies 1290 Infinity II” UPLC (Agilent Technologies, Santa Clara, California, USA) equipped with a Waters “Acquity UPLC BEH” reversed-phase C18 1.7 μm packed column (Waters Corporation, Milford, Massachusetts, USA) was used. Elution of all pyridines was achieved with a gradient with three eluents in the mobile phase. These were a buffer solution of 5 mM NH_4HCO_2 , methanol, and acetonitrile, at a total constant flow rate of 0.5 mL/min. The total time of the chromatographic run was 10 min.

3. Results and discussion

Results are presented in the form of remaining fraction (RF) as function of time (t) and temperature (T). RF of the tested pyridines is defined as $C(t, T)/C(t_0, T_0)$. Analytical uncertainty is deliberately not depicted to facilitate the reading of the figures. Typical standard deviation values (σ) in the determination of the concentration in the triplicate replicas range from 0,04 to 0,08.

3.1. Pyridine

Fig. 1 depicts the RF values of pyridine as function of time and temperature at the different initial pH values.

No clear trend is observable for the RF values of pyridine (Fig. 1) as function of time, temperature, and pH. The variations observed can

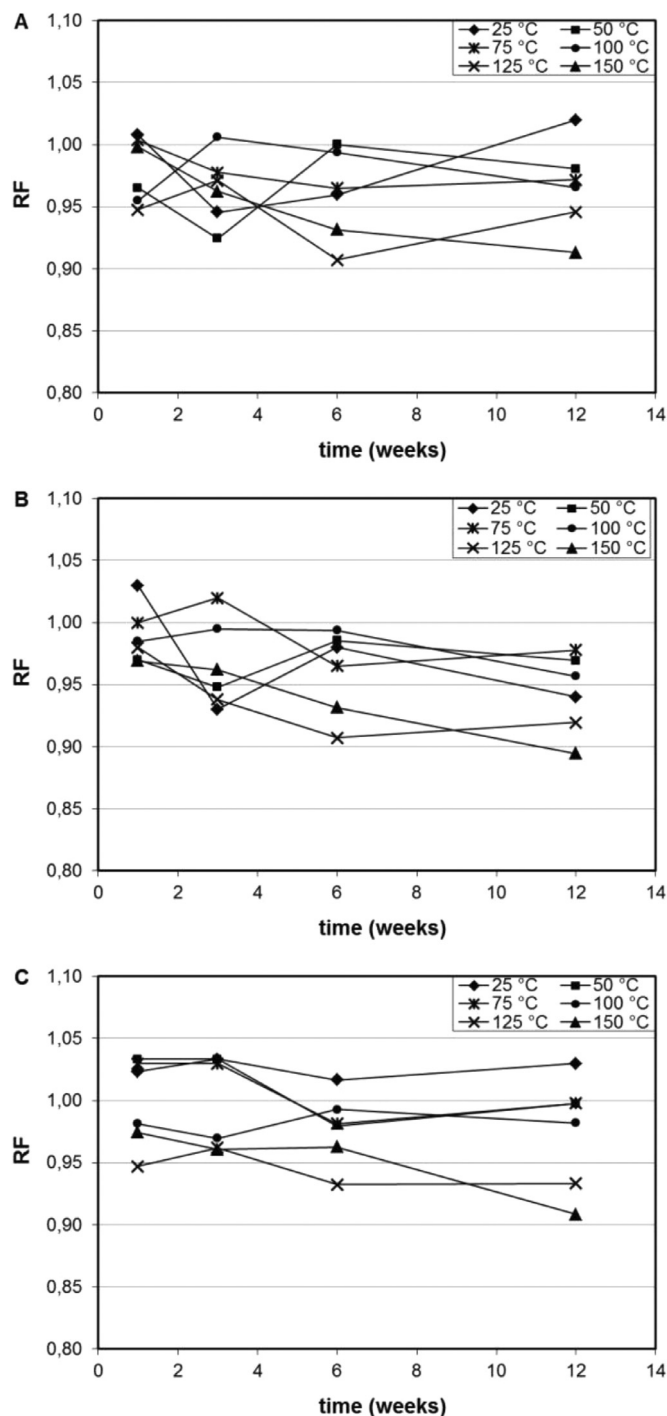


Fig. 1. RF of pyridine as function of time and temperature of incubation. (A) Initial pH 5.5; (B) Initial pH 7.1; (C) Initial pH 8.0 and no rock substrate in any of the samples.

be attributed to analytical uncertainty. Thus, results show that pyridine exhibits the stability required for use as PITT tracer. Mass conservation is critical for this application.

3.2. 2-Hydroxy-6-methylpyridine

2-Hydroxy-6-methylpyridine (results in Fig. 2) is only stable at a temperature of 25 °C. Unsuccessful attempts (not presented in this document) to develop a kinetic model for the degradation of 2-hydroxy-6-methylpyridine were made.

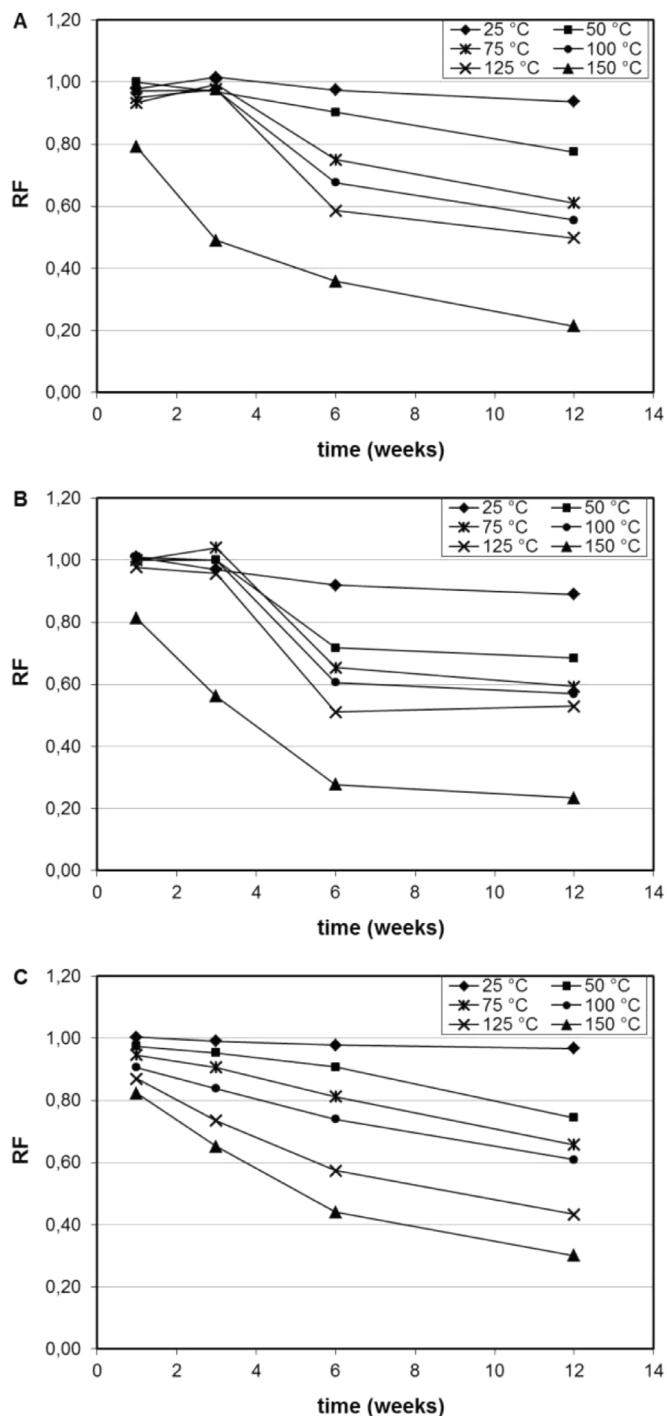


Fig. 2. RF of 2-hydroxy-6-methylpyridine as function of time and temperature of incubation. (A) Initial pH 5.5; (B) Initial pH 7.1; (C) Initial pH 8.0 and no rock substrate.

The decrease in RF values of 2-hydroxy-6-methylpyridine observed at mild temperatures of 50 °C and 75 °C also suggests that this compound may be prone to microbial degradation. The rate of degradation of 2-hydroxy-6-methylpyridine appears to be lower with increasing initial pH values. This effect is particularly noticeable for shorter incubation periods and higher temperatures. As the time of incubation increases, results suggest that temperature becomes the predominant driving force behind the degradation process, and influence of pH becomes less clear. Thus 2-hydroxy-6-methylpyridine is not an effective candidate to consider as PITT tracer. Furthermore, the difficulty in accurately describing

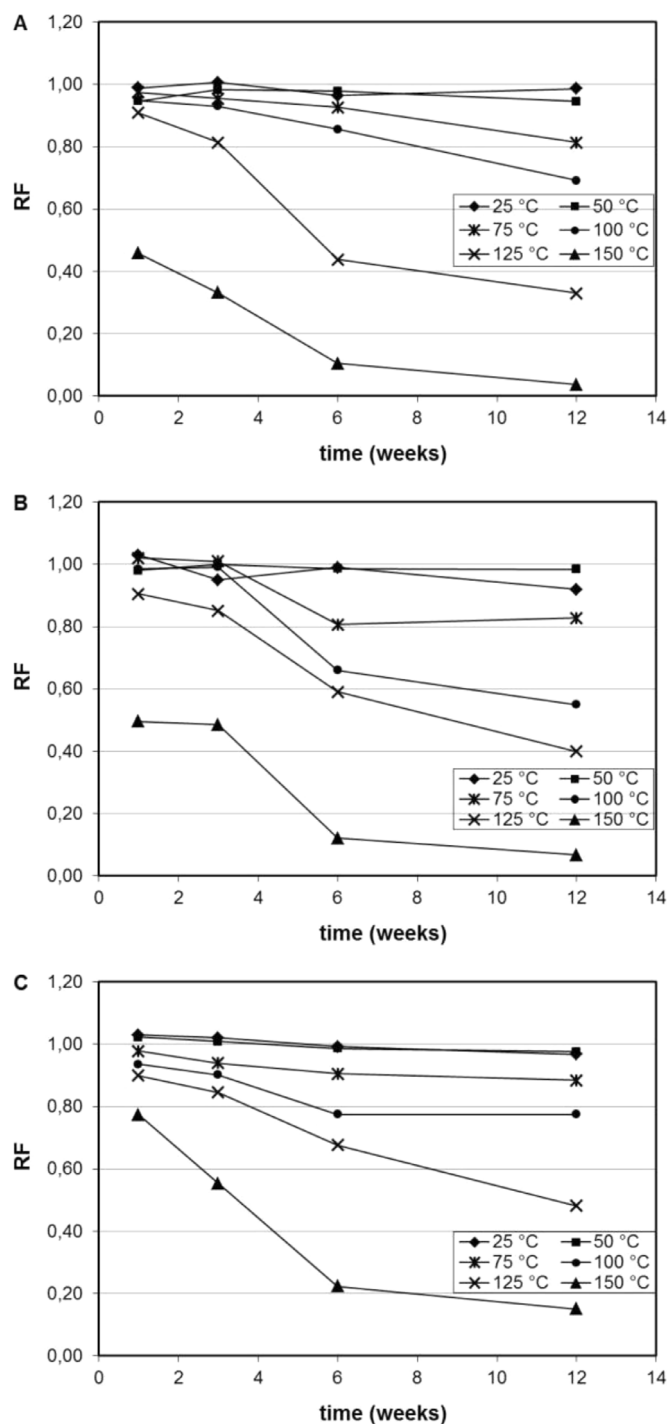


Fig. 3. RF of 3-Hydroxypyridine as function of time and temperature of incubation. (A) Initial pH 5.5; (B) Initial pH 7.1; (C) Initial pH 8.0 and no rock substrate.

the degradation of this compound leads us to conclude that it is most likely inappropriate for any use as tracer.

3.3. 3-Hydroxypyridine/3-Hydroxypyridine

Fig. 3 is stable at temperatures up to 75 °C and 12 weeks of thermal incubation. Microbial activity is unlikely at temperatures where degradation is observed. The use of 3-hydroxypyridine as mass conservative tracer could still be considered in low temperature reservoirs, typically onshore low depth ones, or in environmental applications such as as-

essment of near-surface contaminations by none-aqueous phase liquids (NAPL). Efforts to identify degradation products were unfruitful. The thermally driven degradation of 3-hydroxypyridine appears to be slower at higher pH values, however, this protective effect is not sufficient to compensate for the temperature effect. 3-Hydroxypyridine is unlikely to have any application as tracer in an oil reservoir.

3.4. 4-Methoxypyridine

4-Methoxypyridine degrades at temperatures ≥ 75 °C with no influence from pH (Fig. 4). This suggests a thermally driven degradation mechanism.

The rate at which the degradation of 4-methoxypyridine occurs is not linear. Thus, is not zero order and is possibly concentration dependent. Further treatment of the variation of RF values of this compound was performed with the intention of assessing the accuracy of a first order kinetic degradation model in describing the experimental data. An accurate kinetic degradation model opens the possibility of using 4-methoxypyridine as an active temperature probe to retrieve information about temperature or thermal fronts in reservoirs.

3.5. Influence of rock substrate

Three rock materials (Berea sandstone, limestone and kaolinite) were used as “model” reservoir rocks to evaluate interaction with the pyridines. These materials do not represent all the minerals that would be encountered in an oil basin, but they are representative of the most common sediment constituents and allow for the screening of a wide range of possible effects and interactions with the compounds investigated.

The maximum difference between RF (t,T) values in the experiments with and without rock substrate encountered for each of the tested compounds with their $\pm 2 \sigma$ error bars are shown in Fig. 5.

The maximum differences encountered for pyridine and 2-hydroxy-6-methylpyridine are all within analytical uncertainty. These two compounds have no significant interaction with the rock materials and their use is not limited by the nature of the reservoir rocks.

RF values of 4-methoxypyridine are not significantly influenced by the presence of Berea sandstone and limestone, but dramatically influenced by the presence of kaolinite. In the presence of this clay, the maximum difference in RF values reaches about 0,77. Thus, any field application considering this compound as a possible tracer should be performed with extreme caution. However, this also suggests that this compound can potentially be used to retrieve information about the presence and distribution of kaolinite and perhaps similar clays.

3-Hydroxypyridine is also insensitive to the presence of sandstone and carbonate rock materials and sensitive to kaolinite in a combined effect with pH. Fig. 5 clearly depicts a decrease in the maximum differences between RF in experiments with no rock substrate and experiments with kaolinite with increasing pH values.

Excluding the differences observed at 150 °C, results in Fig. 6 show that there is a clear pH dependence on the interaction of 3-hydroxypyridine with kaolinite.

The lower the pH of the experiment, the larger is the decrease in the concentration of 3-hydroxypyridine in the presence of kaolinite. The pKa value (Table 1) for 3-hydroxypyridinium (the protonated form of 3-hydroxypyridine) is 4.79. Thus, as the pH decreases the concentration of the protonated species increases, which could make it more reactive with the interlayer ions in the kaolinite. The use of kaolinite as catalyst in reactions involving both organic and inorganic compounds has also been reported [29,30]. Thus, another possible explanation for the reduction of the RF of 3-hydroxypyridine with pH is that kaolinite is catalysing a reaction between 3-hydroxypyridinium and one of the metallic cations present in solution.

The possibility that kaolinite is adsorbing either the protonated form of 3-hydroxypyridine or its other possible products is not likely because

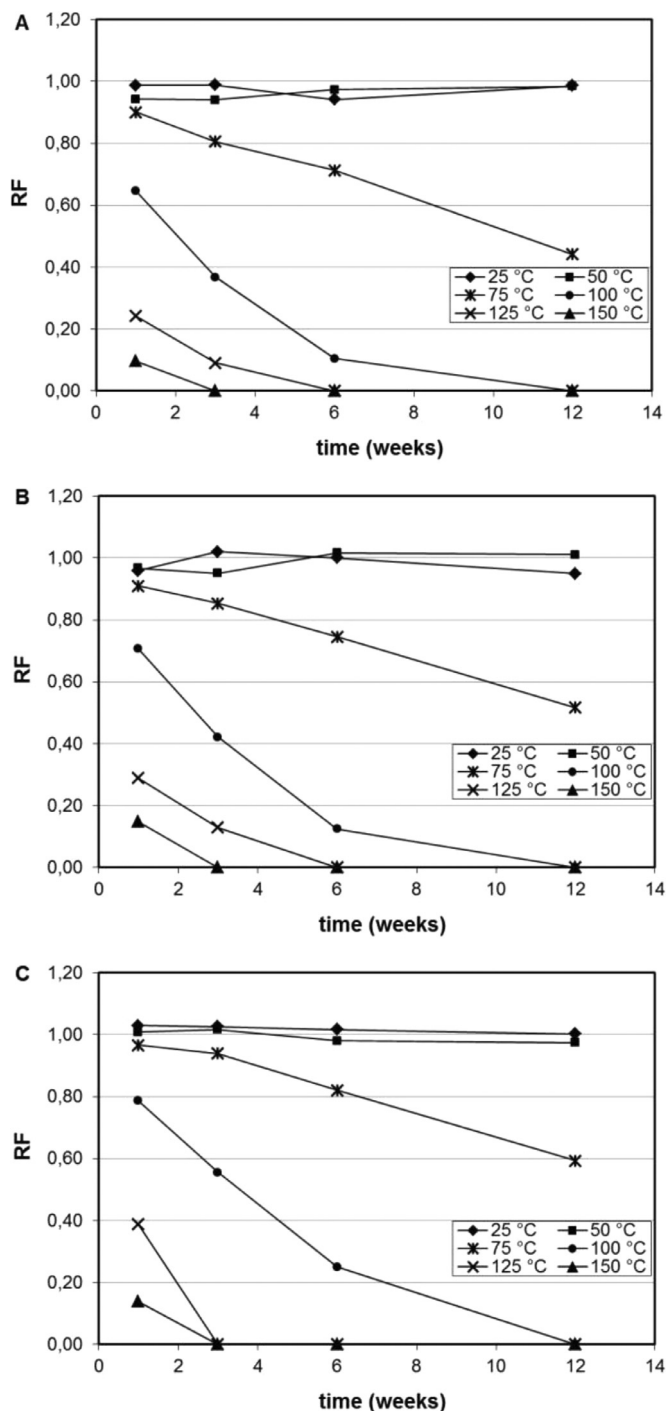


Fig. 4. RF of 4-methoxy pyridine as function of time and temperature of incubation. (A) Initial pH 5.5; (B) Initial pH 7.1; (C) Initial pH 8.0 and no rock substrate.

the adsorption should be a function of temperature. Whatever the cause of reduction of the RF of 3-hydroxypyridine in the presence of kaolinite means that it will not survive long at detectable concentrations in reservoir sediments containing this clay.

3.6. Kinetics of degradation of 4-methoxy pyridine

The thermal stability data of 4-methoxy pyridine was analysed using a first order kinetic model. Changes in C_A/C_{A0} (RF) at 75 °C, 100 °C, 125 °C, and 150 °C are shown in Fig. 7.

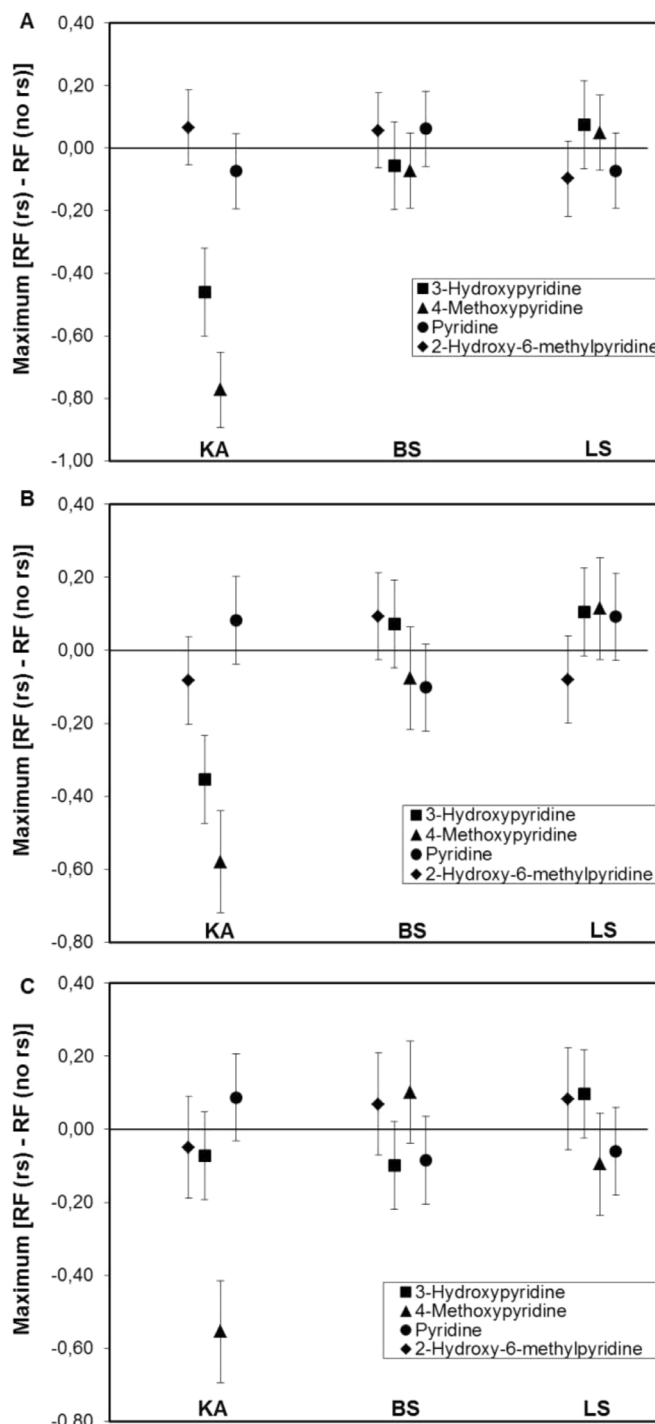


Fig. 5. Maximum differences encountered in RF values between experiments with rock substrate (rs) and experiments without rock substrate $\pm 2 \times \sigma$ in RF (t, T). (KA) kaolinite, (BS) Berea sandstone, (LS) limestone. (A) Initial pH 5.5; (B) Initial pH 7.1; (C) Initial pH 8.0.

The $\ln(C_A/C_{A0})$ in Fig. 7 were linearly regressed as function of time of incubation. For first order kinetics, the apparent rate constant of the reaction (k) (weeks⁻¹) at each temperature is the corresponding slope of the linear regression. With the linearised Arrhenius law [k (T) as function of 1/T] the apparent activation energy of the reaction and the pre-exponential factor (A) were obtained with the result presented in Eq. (1).

$$\ln\left(\frac{C_A}{C_{A0}}\right) = -1,86 \times 10^7 \cdot e^{\frac{-56,4 \times 10^3}{R T}} \cdot t \quad (1)$$

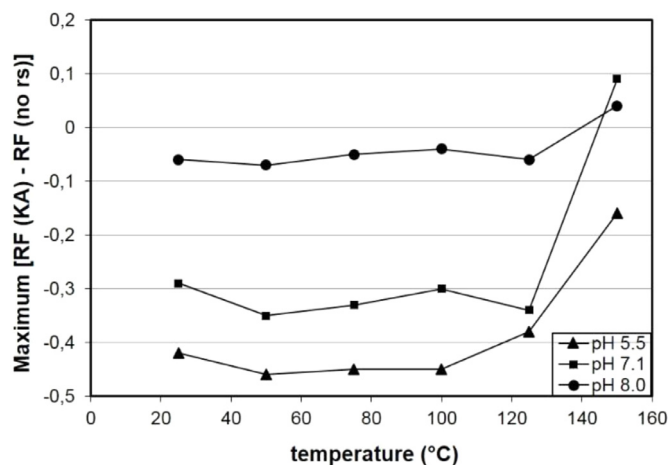


Fig. 6. Maximum differences encountered in RF values of 3-hydroxypyridine between experiments with kaolinite (KA) and experiments without rock substrate (rs) as function of temperature and pH.

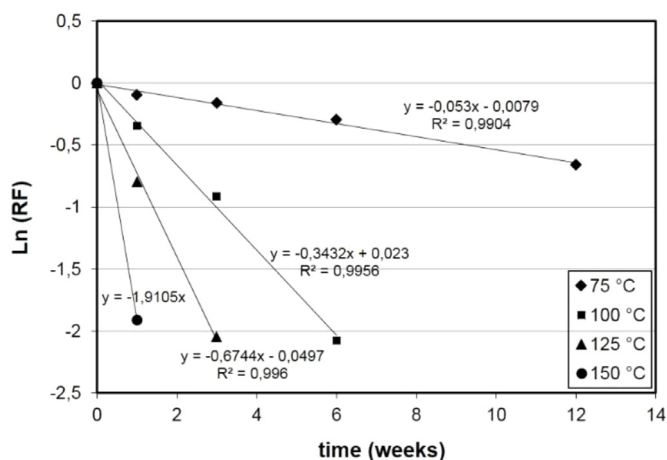


Fig. 7. Ln (RF) of 4-methoxyppyridine as a function of time and linear regressions at each temperature. Initial experimental pH 7.1, no rock substrate present.

The apparent activation energy of the reaction is 56,4 kJ/mol and $A = 1,86 \times 10^7$.

The accuracy of the model was tested by comparing its predicted RF values with the experimental ones. Two additional test samples (2.2) were prepared to obtain RF after 2 and 4 weeks of incubation. Experimental RF values $\pm 2\sigma$ in their determination vs RF values predicted by the kinetic model are presented in Fig. 8.

4. Conclusions

The stability of pyridine, 2-hydroxy-6-methylpyridine, 3-hydroxypyridine, and 4-methoxyppyridine was evaluated at relevant reservoir temperatures (25 °C to 150 °C), pH (5,5; 7,1 and 8,0), with elevated salinity, and in the presence of sandstone, carbonate, and clay materials.

Results show that pyridine is not degraded during the 12 weeks of experiment at all tested temperatures. It is also insensitive to the presence of the rock materials and pH variations. Thus, pyridine appears to be fully stable and can be considered as an active (partitioning) mass conservative reservoir tracer. Since it has been reported as a constituent of crude oilsamples of produced waters from the field must be tested thoroughly prior to the use of this compound.

2-hydroxy-6-methylpyridine is only stable at 25 °C during the 12 weeks of testing. The stability of this compound is somewhat influenced

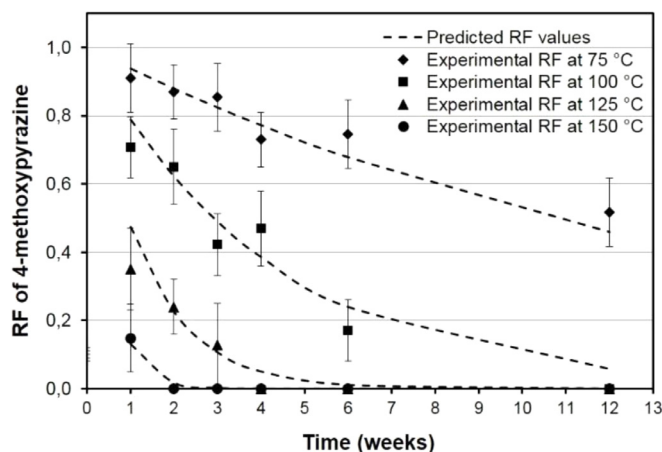


Fig. 8. Experimental vs predicted RF values of 4-methoxyppyridine between 75 and 150 °C. The uncertainty of $\pm 2\sigma$ is not depicted for values under the lower analytical threshold (RF = 0,05).

by pH, becoming more stable at more basic values. This effect is more evident in experiments at high test temperatures and short incubation times. No significant interaction was observed between 2-hydroxy-6-methylpyridine and any of the rock substrates. Results are inconclusive about the nature of the degradation phenomena, and microbial action cannot be excluded. Thus, any possible application based on this compound as tracer in the oilfield is highly unlikely.

3-Hydroxypyridine is fully stable up to 50 °C during 12 weeks of incubation. At higher temperatures, thermally driven degradation with influence from the pH conditions is observed. The rate of degradation decreases slightly as the pH increases. 3-Hydroxypyridine is insensitive to the presence of Berea sandstone and limestone, but a strong effect on its stability is observed under the presence of kaolinite in combination with pH. Results suggest that kaolinite is most likely catalysing a reaction between the protonated species of this compound and a cation present in the bulk, as no temperature effect is observable which would suggest an adsorption phenomenon.

4-Methoxyppyridine is stable for 12 weeks up to 50 °C. Thermally driven degradation of this compound is observed at all other tested temperatures. A strong interaction with kaolinite causes a large decrease of the RF values of this compound in the whole range of temperatures tested. The nature of this interaction is uncertain, as no degradation products were identified. This tracer candidate is not affected by the presence of sandstone, limestone or pH variations (in the studied range) by themselves or in combination with other factors. The degradation of 4-methoxyppyridine is well described by a pseudo first-order kinetic model without participation of other chemical species. The apparent activation energy and the pre-exponential factor of the Arrhenius equation of the thermal degradation were determined. The use of 4-methoxyppyridine to retrieve thermal information from the reservoir is suggested.

Declaration of Competing Interest

The authors have no conflict of interests to declare.

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