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The Development of Advanced Fluorescent Tracers Aimed at Drill Cuttings Labelling and Depth Correlation via Injection with Oil-Based Drilling Mud

Vladimir Khmelnitskiy ¹, Hassan S. Alqahtani ², Hyung Kwak ² and Vera Solovyeva ^{1,*} 

¹ Aramco Innovations, Bld. 1, 9 Varshavskoe Highway, 117105 Moscow, Russia; vladimir.khmelnitskiy@aramcoinnovations.com

² Saudi Aramco—EXPEC ARC (Advanced Research Center), Dhahran 31311, Saudi Arabia

* Correspondence: vera.solovyeva@aramcoinnovations.com

Abstract: Fast and precise geo-steering and geo-navigation upon well drilling are the key parameters for improved well targeting, optimal well placement, and maximal hydrocarbon recovery. To advance geo-steering parameters, we propose a new approach to on-site formation evaluation through the use of fluorescent tracers for drill cuttings tagging according to the depth of origin. Cuttings labelling at the drill bit site is followed by near-real-time drilling depth correlation at the well-head via a camera and AI image recognition systems. To suite the drilling process, the engineered tracers should match to the rheology of the utilized drilling mud. This study was performed to comprehensively investigate the effect of fluorescent tracers on the rheological properties of oil-based drilling mud (OBM) and to determine the optimal quantities of the tracers' addition. We evaluated critical mud characteristics including electrical stability, thixotropic parameters, shear stress, gel strength, plastic viscosity, and yield point as prepared and in the presence of fluorescent tracers at the range of 1 to 20 wt.%. Additionally, the mud's effects on the long-term stability of the fluorescent tracers were assessed via hot-rolling tests in conditions mimicking downhole conditions, with the aim of determining the tags' feasibility for drill cuttings labelling applications. The study also examines the recovery potential of the tracers and their reusability in the drilling process. This investigation provides valuable insights into the potential application of fluorescent tracers for downhole drill cuttings depth correlation which will improve geo-steering works.

Keywords: fluorescent tracers; drill cuttings labelling; hot rolling test; rheology of OBM



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1. Introduction

Since the year 2000, advanced mud logging (AML) techniques have become not only a dream but also a very valuable and fruitful method for real-time well evaluation and resource surveillance via the determination of rock and fluid characteristics from drill cuttings, drilling parameters, and mud gas analysis [1]. Current AML methods of formation evaluation involve enhanced cuttings image acquisition and processing as well as direct measurements on cuttings including grain density, porosity, spectral gamma ray (GR), nuclear magnetic resonance (NMR), X-ray diffraction (XRD), and X-ray fluorescence (XRF) [2]. Nowadays, integrated advanced mud logging units combine several on-site petrochemical measurements and characterization approaches.

Rapid drill cuttings characterization using portable X-ray fluorescence techniques enables [3] the performance of elemental analysis of formation cuttings and converts it into mineralogical composition [4]. X-ray diffraction provides near-real-time mineral analysis [5]. Drill cuttings image acquisition and interpretation allows for the prediction of the electrical parameters of rocks and the lithology classification of reservoirs [6]. These and other petrophysical analyses are very valuable in detecting hydrocarbon pay zones.

However, for accurate geo-steering and well positioning, drilling depth determination is the most important logging parameter.

Conventional mud logging methods that are based on drilling mud hydraulics and the estimation of cuttings lag time are challenging, especially for deviated and horizontal wells where inclination affects hole cleaning. The prediction of cuttings transport mechanisms, lag time, and the efficiency of removal from the wellbore has been the focus of mathematical modelling and correlations accompanied by experimental evaluations on several flow loop systems and field trials [7–9]. Nevertheless, due to the complexity and diversity of reservoir parameters, comprehensive and realistic models are still hard to determine.

Recently, polymeric NanoTags were proposed for drill cuttings tagging according to the depth of their origin. Labelling of the drill cuttings with barcoded polymeric nanoparticles is performed when they are generated at the drill bit site. Comprehensive extraction of tags followed by the pyrolysis-GCMS identification of functionalized polystyrenes [10] or polysaccharides residues [11] allowed for improved drilling depth correlation. Another study describes a recent field test that included automated pulse injection of a sequence of tracers during drilling [12].

Aiming to reduce labor consumption during sample preparation and the analytical workflow, our group developed fluorescent-based tracers for drill cuttings tagging, whose synthesis and laboratory characterization we reported previously [13]. We demonstrated that these tags are practically efficient for drill cuttings labelling applications because they can be easily detected and tracked via a camera with no need for specific sampling procedures [14]. The objective of this study is to assess the feasibility of the use of fluorescent-based tracers for drill cuttings labelling upon drilling by examining their effect on the rheological characteristics, electrical stability, and thixotropic parameters of OBM. Moreover, we evaluated the impact of the tracers' additives on the OBM's shear stress, gel strength, plastic viscosity (PV), and yield point (YP). The hot rolling test, which simulates the conditions of mud and formation cuttings circulation in the well, was used to demonstrate the possibility of the tracers' recovery and reuse.

The rheological properties of the drilling fluid should be kept in the standardized range during the drilling process to allow the drilled mud to maintain proper cuttings transport and borehole cleaning. Novel additives, especially high surface area particles, could affect the stability and effectiveness of the drilling mud functions and thus disturb drilling operations. To avoid such undesirable consequences during field trials, we determined the range of effective concentrations of the tags that are compatible for injections with OBM emulsions and do not impact the rheological behavior of the mud.

2. Materials and Methods

2.1. Materials

We purchased fluorescein, rhodamine B, and HPLC-grade petroleum ether from Sigma Aldrich (St. Louis, MO, USA). We purchased commercial super-absorbent poly-mer (SAP) based on sodium polyacrylate (Prod.# C001B1) from Orbeegun (Moscow, Russia). Oil-based drilling mud was obtained by our laboratory with a density of 1.25 g/cm³. We used all materials and solvents without further purification. We prepared all aqueous solutions with deionized water (18.2 MΩ * cm), Sartorius, Arium® (Göttingen, Germany).

2.2. Method of Drilling Mud Preparation

Oil-based drilling mud (reverse emulsion) was prepared using a Hamilton Beach high-speed mixer with constant stirring. The composition and order of component additions for the OBM are as follows: mineral oil (145 mL) is loaded to the mixer cup followed by the addition of a structural agent organobentonite (hydrophobized clay) (3 g), the mixture is stirred for 15 min at 3 speed; then, we added the primary emulsifier (4 mL) and stirred the mixture for 5 min at 1 speed; following this, we added lime (Ca(OH)₂) (3 g) and treated the mixture for 10 min at 1 speed; next, we added the rheology modifier (1 mL) and stirred the mixture for 5 min at 1 speed; after that, we added a filtration reducer (2.5 g) and stirred

the mixture for 5 min at 1 speed; next, we added calcium chloride (aqueous solution with $\rho = 1.25 \text{ g/cm}^3$) (47.5 mL) under stirring within 20 min with mixing after injection for 10 min at 1 speed; then, we added a hydrophobizing agent (1 mL) and kept stirring for 5 min at 1 speed; and finally, we added a portion of 115 g marble to the slurry to reach a density of 1.15 g/cm^3 and mixed the slurry for 10 min at 1 speed. The resultant OBM was used for the rheology tests as is and with additives of tracers that were placed (1, 5, 10, and 20 wt.%, i.e., 2.4, 12, 24, and 48 g, respectively) to the as-prepared OBM and mixed for 5 min at 1 speed.

2.3. Method of Rheological Properties Determination (PV, YP Gel Strength, and Viscosity)

The tests were performed according to ISO 10414-2:2011 Petroleum and natural gas industries—Field testing of drilling fluids—Part 2: Oil-based fluids [15]. An OBM sample was placed into a six-speed Fann rotational viscometer cup. The temperature of the sample was 49°C . The viscosity curve was obtained by measuring the viscosity at a wide range of shear rates (3 to 600 rpm). We started measurements from 600 rpm. Then, we reduced the rotor speed to 300 rpm and waited until the viscometer scale readings reached a constant value. The measurements of the following points (200, 100, 6, and 3 rpm) were performed the same way. The procedure for the determination of gel strength is described below. Firstly, the speed is set to 600 rpm for 10 s and then the rotor is stopped and the sample is maintained without stirring for 10 s. Then, the viscosimeter is turned on and set to the speed of 3 rpm; the viscosimeter reading is the gel strength (10 s). Next, the sample is stirred at 600 rpm for 10 s, stirring is stopped, and then the sample is maintained without stirring for 10 min. Rotation is started at 3 rpm, and the reading value, which is the gel strength (10 min) is recorded. The unit of gel strength measurement is $\text{lbs}/100 \text{ ft}^2$. The calculation of the plastic viscosity PV in millipascals per second (centipoise) is shown in Equation (1)

$$\text{PV} = R_{600} - R_{300}, \quad (1)$$

where R_{600} is the viscosimeter reading at 600 rpm and R_{300} is the viscosimeter reading at 300 rpm.

The calculation of the yield point YP ($\text{lbs}/100 \text{ ft}^2$) is shown in Equation (2):

$$\text{YP} = R_{300} - \text{PV}, \quad (2)$$

where R_{300} is the viscosimeter reading at 300 rpm.

2.4. Method of Electrical Stability (ES) Determination

The sample of the OBM heated to 49°C was mixed for 10 s using an electrode to counterbalance the parameters. The electrode was installed so that it did not touch the glass, and the electrode surface was fully covered with the sample. Then, a linear change in voltage was started and the electrode was held until the end point was reached and the display provided a steady reading. The resulting ES values should not differ more than 5%. The average of the three ES measurements was then recorded.

2.5. Dye-Loaded Tracers Based on Super-Absorbent Polymer and SiO_2

In the current study, we evaluated two types of dye-loaded tracers that include silica loaded with fluorescein and super-absorbent polymer (SAP) loaded with xanthene dyes. The method of obtaining of tags based on fluorescein-loaded silica (SiO_2 -fluorescein) is described in the work [13]. The second type of tracer based on super-absorbent polymer loaded with either fluorescein or rhodamine B (SAP-fluorescein and SAP-rhodamine) was obtained according to the procedure described in [14].

2.6. Method of Tracers Testing by Hot Rolling

Evaluation of the tracer's stability in the presence of drilling mud was conducted according to ISO 10416:2008 [16] "Petroleum and natural gas industries—Drilling fluids—

Laboratory testing”, chapter 23: “Shale-particle disintegration test by hot rolling”. The mud sample and tracers were placed in a mud-aging cell and sealed, and the cell was then put into a roller oven. The test was conducted in two ways: with and without drill cuttings. The cells were constantly rolled at a temperature of 80 °C for 16 h.

2.7. Assessment of the Fluorescent Intensity and Recovery Possibility of the Tags

The fluorescence characteristics of the tracers were evaluated visually during daylight and under ultraviolet (UV) light. The tracers were separated from the drilling fluid and drill cuttings via sieves after the hot roll test. Certain tracers were also subjected to petroleum ether washing followed by filtration.

3. Results and Discussion

Tracer testing is a well-established technique in the oil and gas industry that commonly includes single well [17] and inter-well tracer campaigns [18]. The use of fluorescent tracers for drill cuttings labelling is a promising new approach for estimation of the formation cuttings depth of origin at the wellhead site. Tagging of the cuttings can be performed via crushing the tracers over the formation with the drill bit. Stained cuttings are transported to the surface with the circulation of the drilling mud. The ease of detection of fluorescent tags enables automated registration of such indicators in near-real-time mode via a camera at the wellhead site. By targeting the injection of the fluorescent tracers with drilling mud, in this work, we investigated the compatibility of the tags with the drilling fluid and determined the range of efficient concentrations of the tracers that can be used without effecting of the drilling procedures. Namely, we examined the effect of the tracers’ additives on the rheological characteristics of the oil-based drilling mud, electrical stability, and thixotropic parameters as well as the effect on shear stress, gel strength, plastic viscosity, and yield point.

Comparison and evaluation of two types of tags were performed in the current study. We examined the effects of silica loaded with fluorescein and super-absorbent polymer loaded with xanthene dyes as tracer additives in 1 to 20 wt.% to the drilling fluid. These tags differ by particle size and the chemical nature of the loaded matrix, where tracers based on fluorescein-loaded silica possess a high porosity and surface area with dimensions of 60 to 200 µm whereas tracers based on SAP are about 1.5 mm-diameter low-surface area spheres.

3.1. Rheological Properties (PV, YP Gel Strength, and Viscosity)

The physical size and high specific surface area of additives (in our case—tracers) can have a significant impact on the rheological behavior of drilling fluids [19]. To evaluate this phenomenon, the SiO₂- and SAP-based tracers were added to the drilling solution at concentrations of 1%, 5%, 10%, and 20%, and their effects on various rheological parameters were measured as a function of quantity of the tracer additives. We performed an assessment on the influence of the tracers’ additives on the rheology of oil-based drilling mud (OBM) according to the ISO 10414-2:2011 standard [15]. It is crucial to ensure that the presence of tracers in the mud formulation would not interrupt the well drilling progress, and thus, the presence of tracers should not affect the mud rheology over the standard recommended range. Therefore, our investigation provides insights into the range of efficient concentrations of tracers that can be safely added into the drilling mud formulation without affecting its properties.

3.1.1. Influence of Tracers’ Additives on the Viscosity of the OBM

The viscosity of the OBM samples was recorded with a six-speed Fann rotational viscometer at 3, 6, 100, 200, 300, and 600 rpm according to ISO 10414-2:2011 [15]. The experimental data and the flow curve for each OBM fluid are presented in Figure 1, Table S1. It was noted that additives of dry superabsorbent-based tracers added to OBM did not affect the viscosity of the OBM at the entire tested concentration range (up to 20 wt.%).

High surface area SiO_2 -based tracers affected the plastic viscosity of the OBM the most—up to 88% at the maximal loading of 20 wt. %

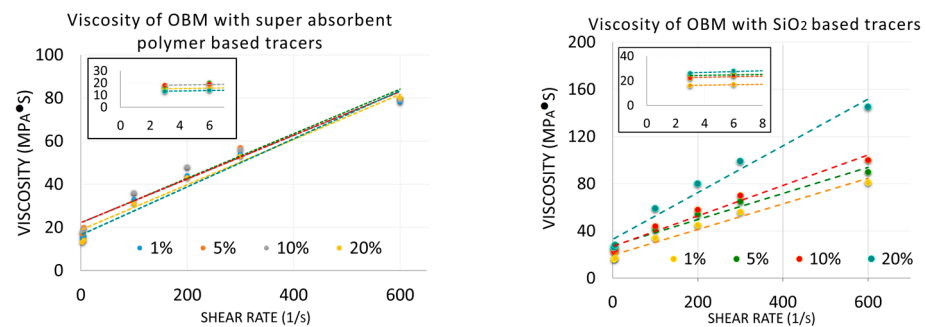


Figure 1. Rheology curves of the OBM with tracers.

3.1.2. Influence of the Tracers' Additives on the Plastic Viscosity of the OBM

Plastic viscosity (PV) is an important rheological characteristic that reflects the resistance to flow due to inter-particle friction. Plastic viscosity (PV) depends on the amount of solids in the mud, the size and the shape of these solids, and the viscosity of the liquid phase. The flow resistance is a result of friction between the liquid undergoing deformation under shear stress and the solids present in the drilling mud. PV is a parameter of the Bingham plastic model and is the slope of the shear stress/shear rate line above the yield point. Experimentally, the value of PV resulted from subtraction of 300 rpm readings from 600 rpm readings of the Fann viscosimeter [20]. Figure 2 summarizes the results of the PV measurements depending on the amount of added fluorescent-based tracers Table S1.

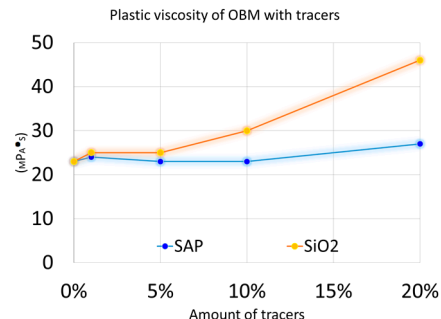


Figure 2. Correlation between the plastic viscosity values and the tracer concentration.

According to the graph in Figure 2, the PV is nearly constant at low concentrations of SiO_2 - and SAP-based tracers; however, with the increasing concentration of SiO_2 -based tracers over 5 wt. %, the PV increases continuously. The plastic viscosity of emulsion with a high concentration of SiO_2 -based tracers rises rapidly, with the total increase reaching up to 100 percent of the initial value at 20 wt. % of the additive. Notably, high-surface area silica-based tags affect the PV much more compared to SAP-based tags which demonstrated very low impact on PV even at 20 wt. % loading. The negative effect of the high PV value of the drilling mud is in the low performance of the cuttings transport to the surface.

3.1.3. Influence of the Tracers' Additives on the Gel Strength and Yield Point of the OBM

The yield point is one of the rheological properties that measures the solid suspending capacity of the mud. The yield point is the yield stress extrapolated to a shear rate of zero [20]. The yield point values increase with the concentration of SiO_2 -based tracers (Figure 3).

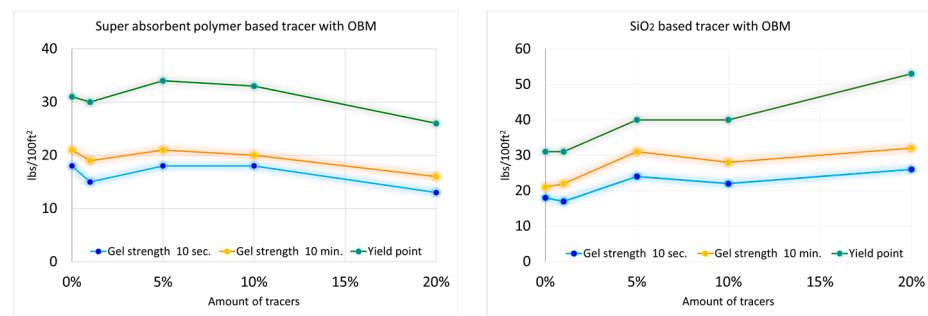


Figure 3. Correlation between the gel strength and yield point data and the tracer concentration.

Gel strength is a measure of interparticle forces and indicates the gelling that will occur when circulation is stopped. This property prevents the cuttings from setting downhole. High gel strength values indicate a higher demand of pump power to circulate the mud and, therefore, are undesirable. On the other hand, low values of gel strength may cause well clogging and pipe sticking due to poor cuttings holding. Commonly, the recommended value of gel strength for OBM should be between 3–40 lbs./100 ft². While the allowed limit was exceeded for SiO₂-based tracers, it was not exceeded for SAP-based tracers. It can be concluded that concentrations of SiO₂-based tracers should not be more than 5%, and SAP-based tracers have no significant effect on the yield point. The results of the gel strength and yield point evaluation experiments are presented in Figure 3, Table S1.

The experimental data of the gel strength measurements represented in the graphs in Figure 3 for both types, SiO₂- and SAP-based fluorescent loaded tracers, match the recommended values across the entire concentration range from 1 to 20 wt.%. The values of yield point for the SAP-based fluorescent tags were almost constant with the increase in the tag concentration which means that additives of SAPs do not influence the resistance of the OBM to the initial fluid flow much. However, the additives of SiO₂-based fluorescent tags gradually increase the YP values upon increasing concentrations of tags. Such an effect should be monitored as a precaution since it can cause frictional pressure losses upon mud applications.

3.2. Influence of the Tracers' Additives on the Electrical Stability of the OBM

To evaluate the stability of the OBM emulsion towards phase separation in the presence of tracer additives, we performed measurements of the electrical stability (ES) of the oil-based drilling mud as is and in the presence of tracers (from 1 to 20 wt.%). The ES of the OBM is a parameter characterizing the degree of dispersion of emulsified water and the strength of the stabilized layer of the emulsifier which is an indirect parameter characterizing the aggregative stability of emulsions. It is determined by the magnitude of the voltage of the electric current required for the electrical breakdown of the OBM layer placed between the electrodes. Low values for the electrical stability of the oil-in-water emulsion are likely to cause fast phase aggregation and sedimentation of the emulsion.

The results of the electrical stability experiments are presented in Figure 4, Table S1. The baseline in this graph is the measurement of the ES value of the pure OBM without tracers; the minimal value represents the lowest ES readings, which allows for the use of OBM for drilling operations. The ES readings of the OBM with additives of SAP-xanthene dye-based tracers exhibited stable permissible deviation of the ES values around the baseline OBM readings upon the increase in the tracer concentration. Contrary to that, increasing the concentration of SiO₂-based tags in OBM above 10 wt.% leads to the diminishing of the electrical stability of the emulsion below the minimal value.

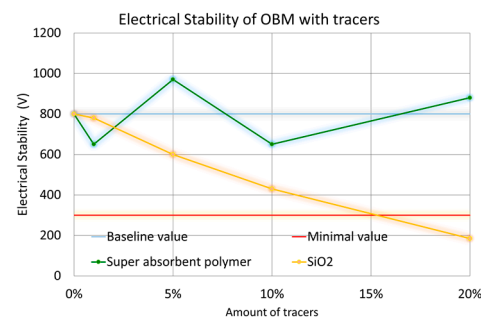


Figure 4. Correlation between the electrical stability data and the tracer concentration.

The acquired observations allowed us to conclude that in the case of tags based on SAP loaded with xanthene dyes, there were no major effects on the rheological characteristics of the drilling fluid upon the addition of materials up to 20 wt.%. However, upon evaluation of the influence of SiO₂-based tracers on the OBM's properties, a significant deterioration in its properties was found after the addition of tags over 5 wt.%. Thus, further evaluations of the tags were performed only for the tracers based on SAP loaded with xanthene dyes.

3.3. Assessing the Fluorescent Tracers' Behavior: Hot Rolling Disintegration and the OBM-Exposed Recovery Test

The hot rolling test mimics the dynamic conditions of downhole particle circulation. During the standard test, the tracers and drill cuttings suspended in the OBM are exposed to constant rolling at 80 °C for 16 h. Upon exposure of the tags to the standard test conditions, SAP-based tracers loaded with xanthene dyes demonstrated excellent thermal and mechanical stability with no decrease in fluorescence intensity [14]. This observation led us to examine the possibility of the recovery and re-use of the tracers. In this work, we evaluated the long-term stability of tracers under the shale particle disintegration hot rolling test conditions using the same tracers repeatedly Table S2.

First, we extended the duration of the hot rolling test by 24 h. Figure 5 demonstrates the visual appearance of the tracers under UV light after one-day exposure to the conditions of the hot rolling test. It is worth noting that the recovered tags remained intact and retained their fluorescence. These tracers were recovered and further utilized for the extended hot rolling tests.

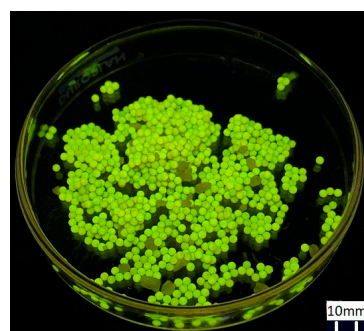


Figure 5. Tracers' appearance under UV light after one-day exposure to the hot rolling test conditions.

Next, we extended the duration of the hot rolling test to 5 days of continuous tumbling. Images of the tagged cuttings under day light and UV light after 5 days of exposure to the hot rolling test conditions are represented in Figure 6. These images confirm the excellent stability of the tags under conditions mimicking dynamic mud circulation and the ease of the tags' detection on drill cuttings.

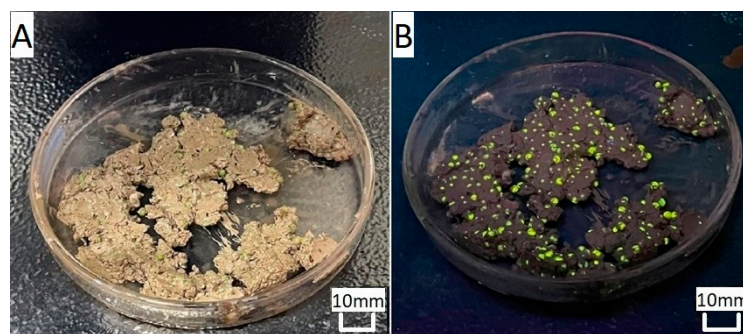


Figure 6. SAP-fluoresceine-based tracers after exposure to the hot rolling test conditions for 5 days: (A) under day light and (B) under UV light.

We further examined the stability of the tags upon long-term exposure to the OBM solution at RT. Figure 7 represents the appearance of pink SAP-rhodamine B-based tracers under UV light recovered from the drilling mud after 4 months of exposure to OBM. Most tags are still visually detectable and retained their fluorescence, shape, and size, even though some of the particles are blurred.

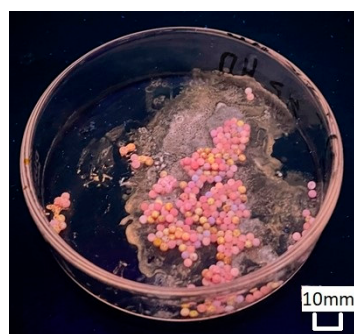


Figure 7. SAP-rhodamine B-based tracers recovered after exposure to OBM for 4 months.

It is important to note that both silica- and SAP-based tracers loaded with xanthine dyes demonstrated excellent stability and minimal dye leaking upon exposure to the oil-based drilling mud even upon long-term tumbling at elevated temperature. Polar structures of the dyes incorporated into the polar carrier matrices bearing hydroxy- or carboxylate-functional groups could be efficiently entrapped and prevented from leaking into non-polar OBM media due to electrostatic interactions.

Notably, small, micron-sized SiO_2 -based fluorescent-loaded tags highly influence the drilling mud rheology; however, they are less detectable than higher mm-sized sodium polyacrylate-based fluorescent tracers (SAPs). The incorporation of the fluorophores into visible size SAP assemblies allows for a reduction in the dye's consumption and prevents it from dissipating upon circulation within excessive amounts of fluid. Furthermore, incorporation of the diluted dyes into the polymeric matrix network prevents self-quenching of xanthene dyes' fluorescence due to high concentrations of fluorophores.

Additionally, polar fluorescent loaded tags are limitedly wettable with non-polar oil-based mud slurry, and due to this, the surface of the tags remains almost clean after exposure to OBM. Thus, the obtained visible size fluorescent tags are readily detectable in UV light visually or via a camera and could be further distinguished and quantified with image recognition systems.

To conclude, the results obtained in the current work indicated that SAP-based fluorescent-loaded tracers could withstand exposure to downhole conditions in OBM up to 4 months with no significant changes in fluorescence intensity and no signs of degradation. Long-term experiments confirmed that engineered tags could be recovered and reused after exposure to conditions mimicking downhole media. The tags remained intact

and able to label the formation particles with strong UV colored labels detectable even without any additional purification of the cutting's mixture. Due to the better compatibility of SAP-based fluorescent-loaded tags with OBM rheology and because of the ease of their detection and analysis, we conclude with considering these materials as the most promising tracer-candidates for drill cuttings tagging according to the depth of origin.

4. Conclusions

In the current work, we evaluated the influence of tracers' additives on the rheology of OBM and proved the compatibility of tracers with OBM functions. Based on the performed experiments, we estimated the efficient concentrations of fluorescent-loaded tracers that are compatible with injection in OBM for applications of drill cuttings tagging. For SiO₂-fluorescein tags, the concentration should not exceed 5 wt.%, whilst for SAP-xanthene dyes-based tags, the applicable concentration could be up to 20 wt.%.

Furthermore, we discovered that SAP-based fluorescent tracers are highly stable under conditions mimicking downhole mud circulation media and could be recovered from the OBM without the loss of fluorescence and reused. Our findings suggest that engineered fluorescent-loaded tracers are a viable opportunity for the improvement of geo-steering operations via drill cuttings labelling according to the depth of the tag's injection with OBM.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/pr11113197/s1>.

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