Microwave separation of persistent oil emulsions

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Abstract. At present, the development of more effective methods for preparing such oils requires special attention due to the growing share of hard-to-extract high-viscous hydrocarbon reserves. The study examined the effectiveness of the thermochemical method and the method combining microwave radiation intended to separate high-viscous emulsions resulting from the thermal methods to increase oil recovery. The results of the research justify the need to use a complex impact aimed at separating persistent oil emulsions.

1. Introduction
Modern oil production has the increasing share of hard-to-recover hydrocarbon reserves [1–3], in particular, heavy and high-viscous oil with a viscosity of 30 mPa · s and higher. Such hydrocarbon resources are several times higher than the reserves of traditional oils and therefore are considered as a reserve for oil production. In Russia, hard-to-recover oil reserves are estimated at more than 7 billion tons. In this regard, it is necessary to pay attention not only to efficient production methods [4–7] which are currently quite complex and require significant costs but also to the issues of collecting and preparing such oils.

The work aimed to study the effects of microwaves on persistent oil emulsions. Meanwhile, the authors stood following tasks: to determine the initial content of mechanical impurities and the water content; to select empirically the time and power of exposure to eliminate the negative effects of microwaves; in a centrifuge, to treat hydrocarbons with a saturated aqueous solution of sodium chloride in different proportions to create favourable conditions conducive to more efficient separation of hydrocarbon mixtures.

2. Methods and materials
The study used a comparative analysis of the effectiveness of the separation of persistent emulsions by the thermochemical method followed by centrifugation and a method combining microwave irradiation (in different operation modes of the plant).

3. Results and discussion
For the preparation of heavy high-viscosity oils, in some cases, technologies include the use of hydrocarbon solvents (light oils, petroleum naphtha fractions, condensate, etc. with long-term sludge) at a temperature of at least 60 ° C. Solvents do not exclude the use of other known processing
methods. The most common method is the treatment of high-viscous oil with a diluent based on the oil fraction and a demulsifier, and the oil is heated before the introduction of the demulsifier.

Another option is to introduce a hydrocarbon solvent containing oil-soluble demulsifier and ballast water into the oil. Oil processing occurs at intensive mixing with the introduced reagents [8].

According to the authors of [9], the combined method, including mixing with light oil in a vast range of ratios (from 1:1 to 1:100), long-term sludge (7–24 h), elevated processing temperature (up to 80 °C), increased consumption of effective demulsifier (in particular, dissolvan 4411–250 g/t) helps to successfully destabilize and destroy high-viscous emulsions resulting from the thermal methods to increase oil recovery, the content of solids in which does not exceed 1%. The authors consider it effective to dose a large amount of demulsifier into the intermediate layer followed by feeding the intermediate layer at the beginning of the process, into the crude oil.

Methods using electric, centrifugal and magnetic fields are suitable for the preparation of heavy oils containing a significant number of solids [10]. However, these methods did not receive proper development and did not find wide application in the practice of preparing slop oils. There are combined methods for processing slop oils including treatment with specially developed reagent compositions, ballast (drainage) waters at temperatures of 60–80 °C. The compositions include demulsifiers, wetting agents and other chemicals in various ratios. Combined methods can successfully destroy slop and pit emulsions containing up to 12% of mechanical impurities by weight.

The methods used today for breaking oil-water emulsions are based on the contact heating methods and chemical additives (demulsifiers). Contact thermal methods have excessively high-energy costs, and chemical additives can pollute wastewater and processed products. An alternative is contactless microwave demulsification technology patented more than two decades ago. Microwaves have high penetrating power and act on water, oil and interfacial films selectively providing a separate supply of energy. It results in a significant increase in the phase separation rate and a decrease in power consumption compared to conventional heating methods [11, 12].

Studies conducted in various countries have established the high efficiency of microwave energy in various fields of economic activity: industry, agriculture, medicine, etc. The search for new applications of microwave processing methods aimed at increasing efficiency and expansion of the applications of ultra-high frequency fields occupying the frequency range from 300 MHz to 300 GHz continues successfully.

An electromagnetic wave penetrates a material and interacts with charged particles. The combination of such microscopic processes leads to the absorption of field energy in the object. The quantum theory helps to obtain a full description of this effect. However, the successful design of microwave devices requires only to the macroscopic properties of the material medium described by classical physics.

Depending on the location of the charges, the molecules of the dielectric medium can be polar and non-polar. In some molecules, the location of charges is so symmetrical that their electric dipole moment is zero without an external electric field. Polar molecules have some electric dipole moment even without an external field. Applying external electric field results in polarization of nonpolar molecules and breaking the symmetry of the arrangement of their charges, and the molecule acquires some electric moment.

Under the influence of microwaves on oil sludge, the temperature of the latter increases two times faster than the temperature of water and 10–20 times faster than the temperature of solid rocks. In this case, the rate of thermal action process on the system is much higher than with classical heating methods. The microwave action excites the dipole rotation of medium molecules in the presence of powerful intermolecular bonds resulting in hysteresis between the applied field and the induced response, and the stored energy is released during relaxation in the form of heat. Theoretically, such treatment should lead to more efficient separation of high-viscous mixtures into their constituent components: hydrocarbons, water, solids.

The studies of the microwave effect on various samples of persistent oil emulsions showed that the main energy is absorbed by the aqueous phase concentrating in globules coated with an armour shell.
It results in arising volumetric heat sources in water globules and their intense heating leading to the destruction of the armour shell [11, 12].

However, the burst temperature of the shell varies depending on the strength and thickness of the armour. Low temperatures are sufficient to rupture thin shells. As a result of the destruction (melting) of the shells, the droplets merge, and the emulsion exfoliates [12].

With significant strength and thickness of the shell, the burst temperature and pressure inside the shell can be so large that the shell ruptures and a single drop of water produces many small droplets. It leads to the formation of a finely dispersed even more stable system, i.e. there is a negative effect of microwave exposure.

For laboratory studies of the microwave effect on hydrocarbon mixtures, we used a Panasonic Microwave Oven (2.45 GHz, 900 W) household microwave oven with an adjustable output power range. The object of the study was samples of persistent oil emulsions. A preliminary analysis of the samples showed that close (almost identical) densities of the dispersion medium and the dispersed phase are a significant factor in the stability of the studied objects. We determined the initial content of mechanical impurities by GOST 6370-83 "Petroleum products and additives. Method for determination of mechanical impurities” and water content by GOST 2477-65 “Oil and oil products. Methods for determining the water content”: sample No. 1 the water content is 28% of the mass, mechanical impurities are 3.1% of the mass; sample No. 2 the water content is 27% of the mass, mechanical impurities are 3.7% of the mass.

Studies on the destruction of the samples used the same conditions to obtain comparable results. We took and dispersed samples as quickly as possible to prevent them from cooling. The initial temperature of all experimental samples was 75 °C. Then we experimentally calibrated the exposure time and power so that after processing the temperature of the samples did not exceed 90 °C to avoid negative effects of the microwaves. The exposure time was 50 seconds. The heating power was 50% of the nominal (Figure 1).

To create favourable conditions for the separation of hydrocarbon mixtures in a centrifuge, we performed the processing of hydrocarbons with a saturated aqueous solution of sodium chloride (brine) in different proportions. It required the saline because the density of the aqueous phase in the process of demulsification increases, and thereby contributes to a greater separation of water from oil. Centrifugation was carried out at sample temperatures of 90 °C and speeds of 2600 rpm. The average value for the two samples allowed to determine the water content, solids (Figs. 2, 3).

Figure 1. Photograph of separation by centrifugal distillation for 10 minutes of the sample after microwave exposure
The study used a comparative analysis of the effectiveness of the separation of persistent emulsions by the thermochemical method followed by centrifugation and a method combining microwave irradiation (in different operation modes of the plant).

Laboratory studies have shown that the sample heating to 90 °C by the usual method and centrifuging for 10 minutes only results in the initial separation of water. At the same time, processing the sample with microwaves allows reaching a temperature of 90 °C and leads to a complete separation of the sample phases.

The centrifugation time ranged from 2 to 10 minutes in the experiments.

The viscosity of the diluted hydrocarbon was 28 MPa•s, and the viscosity of the initial hydrocarbon was 75 MPa•s at temperatures of 90 °C. It was found that the weighting of the aqueous phase leads to an intensification of the separation of hydrocarbon mixture with water (Figure 3).

As a result of the experiments, we recorded the separation of samples into hydrocarbon, water and solid phases.

Experimental data confirmed that microwave exposure promotes a deeper separation of obsolete emulsions in addition to other methods (mixing to separate the densities of different phases, heating, centrifugation).

4. Conclusion
Analysis of the results showed that the use of microwaves reduces the cost of reducing the viscosity of resistant emulsions to effectively separate them. The increase in the water phase with using microwave action increases the demulsification and reduces the separation time in a centrifuge.

Comprehensive preparation with microwave exposure allows the separation of persistent emulsions, including slop oils, intermediate layers and other oil sludges since the separation rate of the hydrocarbon mixture increases due to the destructive effect of electromagnetic fields on the armour shells of water droplets of emulsions.

The main advantages of today’s microwave installations compared to traditional technologies are the ecological purity of the technological process, its simple mechanization and automation, a significant reduction of technological time for the creation of the final product, reduction of energy and economic.

Figure 2. Photograph of separation by centrifugal distillation for 10 minutes of the sample and the addition of 15% saltwater after microwave exposure

Figure 3. Photograph of separation by centrifugal distillation for 10 minutes of the sample and the addition of 20% saltwater after microwave exposure
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