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Continuous monitoring of residual water content in boiling water-hydrocarbon emulsions during thermomechanical dehydration

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ABSTRACT

Significant waste resources are generated in the form of water-oil emulsions. These emulsions cannot be effectively destroyed on an industrial scale by traditional methods that rely on the settling of the aqueous phase, and therefore, they accumulate in large quantities. Thermomechanical dehydration, based on the evaporation of the water phase, presents a promising process for recycling such waste. However, within the framework of thermomechanical dehydration, the issue of optimizing energy costs for heating raw materials and controlling the water content in the product arises. Standard methods of determining water content under the boiling conditions of highly stable water-hydrocarbon emulsions are characterized by low efficiency, as they require constant sampling and the involvement of additional equipment and personnel. Consequently, this presents a challenge in predicting and creating an automated thermomechanical dehydration process. Therefore, dynamic curves depicting changes in the water content of these emulsions, depending on the temperature of the boiling liquid, have been obtained. It is proposed to determine the rate of temperature increase (dT/dt) of the boiling emulsion for continuous, real-time monitoring of the residual water content and for recording the moment of complete dehydration. Achieving a boiling emulsion temperature of 130–170 °C (or higher) and/or the rate of temperature increase from 3.0 to 5.5 (or above) indicates the complete dehydration of the emulsion. The proposed method can be implemented in any industrial or laboratory-scale unit for thermomechanical dehydration without significant capital costs. It is based on the use of simple devices consisting of temperature sensors and a computing unit for determining the temperature and rate of heating.

1. Introduction

Along with highly viscous natural hydrocarbons, the possibility of utilizing secondary feedstock to obtain potential products with a high market value presents an urgent problem nowadays. For example, cutting fluids, which are actively used as lubricants for processing metals and alloys, become contaminated with various impurities, are subject to biodegradation, and ultimately lose their technological properties [1]. The decomposition of the cutting coolant emulsion is accompanied by the formation of sludge that cannot be subjected to traditional dehydration methods. The specific hydrocarbon composition of the coolant (35% to 45% of low-boiling compounds (initial boiling point −160 °C), 55% to 65% of 160–450 °C fraction) allows us to consider it as a potential component of boiler fuel [2].

Intermediate layer emulsions and oil sludges are consistently formed at various stages of crude oil preparation (settling tanks), processing (heavy pyrolysis resin at the stage of pyrogas quenching and primary fractionation *etc.*), transportation (cleaning railway tanks for oil) and storage [3,4]. Consequently, large volumes of oil sludge accumulate, environmental pollution occurs, and quantitative losses of valuable hydrocarbon raw materials are observed, along with a decrease in the working volume of the equipment.

Conventional dehydration methods (settling, centrifugation, extraction, electrical dehydration, *etc.*) cannot be applied to this type of feedstock, as they deal with highly stable polydisperse water-hydrocarbon emulsions that have relatively close densities of the water and hydrocarbon phases; therefore, methods based on settling become unsuitable for such emulsions. In contrast, the thermomechanical dehydration (TMD) technique has proven to be an efficient method for dehydrating the aforementioned emulsions under mechanical agitation through evaporation [5–9]. However,

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within this technology, there is an urgent need to organize continuous, real-time monitoring of the water cut in the boiling phase. Depending on the water content of these emulsions and their origin, the adhesion strength between water and hydrocarbons can vary significantly; that is, the initial and final boiling points of the aqueous phase from such emulsions differ from the boiling point of water (100 °C) and span a wide range. Obviously, within the framework of thermomechanical dehydration, the issue of optimizing energy costs for heating raw materials and controlling the water content in the product arises. When choosing a particular water-cut control method, the key indicators are the simplicity and low cost of the equipment used, as well as the absence of a need for constant sampling.

In general, the water content in petroleum products can be determined by physical or chemical methods [10–14]. Chemical methods are divided into qualitative and quantitative. Qualitative methods are designed to establish the presence of water in petroleum products by detecting changes in the color of chemicals or in the pH value of an aqueous solution [15]. Quantitative water content in petroleum products can be determined using volumetric, titration, and calorimetric methods [16–19].

Volumetric methods are based on measuring the volume of gas (hydrogen, acetylene, ammonia, *etc.*) released when water in petroleum products interacts with chemical reagents (hydrides, carbides, metal nitrides, amides, *etc.*) or by measuring changes in pressure. Titrometric methods offer greater accuracy than volumetric methods and are used to measure small amounts of water in petroleum products. Karl Fischer reagent is often employed; however, it is toxic, unstable during storage, and undesirable for use at low water levels in oil [20]. Additionally, petroleum contains various sulfur compounds, and motor oils have additives that can react with Karl Fischer Reagent, which reduces the accuracy of water content measurements [21]. Incomplete solubility of the sample during Karl Fischer titration and adsorption of compounds by the electrode also negatively impact the results obtained [22–24].

Calorimetric methods determine the amount of heat released during the exothermic interaction of chemical reagents with water (GOST 26378.1—2015. "Used petroleum products. Method for determination of water"). However, the reagent must react with water to release a sufficient amount of heat and must be chemically inert with respect to other components. As the water content decreases, it is necessary to use reagents that provide a greater thermal effect [25,26].

Electrochemical methods (coulometric, conductive, potentiometric, and polarographic) are also used to determine water content [27–29]. The most common coulometric methods rely on a water-sensitive reagent's ability to form cells on an electrode, and on measuring reaction products during electrolysis. During electrolysis, chemical and electrochemical processes occur simultaneously, allowing for continuous determination of water with high resolution and sensitivity (up to 0.001%). However, a disadvantages of this method is the requirement for pre-dried inert gas for water extraction [19].

Within the framework of physical methods, water content is determined without altering its molecular state using optical, chromatographic, electrical, distillation, and other methods. Qualitatively, the presence of water is detected through specific properties of materials, such as changes in their geometric dimensions, luminescence under ultraviolet light in the presence of water, and the formation of bubbles when exposed to a hot surface, as observed in the crackle test, *etc*. [10].

Among the quantitative methods for determining water content, gravimetric and dielcometric methods are commonly used. Centrifugation measures only the suspended water, and its effectiveness can depend on temperature [23]. One method involves analyzing the mass loss of a sample under thermal influence. Although this method is easy to implement, it is time-consuming; furthermore, thermal dehydration products and volatile substances can negatively affect the accuracy of the analysis results [30].

The standard Dean-Stark method (ASTM D95—13(2018)) is based on azeotropic distillation. This method is labor-intensive and time-consuming, requiring additional reagents-solvents. According to ASTM D95—13(2018), it reproducibility and repeatability significantly decreases when the water cut is less than 0.5% [31].

Water content can also be determined by various methods, including magnetic permeability [32], dielectric strength [33], dielectric loss tangent [34], infrared (IR) methods [35–37], Raman spectroscopy [38], electrochemical impedance spectroscopy [39], nephelometry [40], photocolorimetry [41], chromatography [22], gas chromatography-mass spectrometry (GC-MS) [42–44]. These devices are widely known and come in different designs. However, they require periodic calibration with reference fluids and specialized analytical equipment. The method based on gas chromatography is considered more effective; however, detecting water requires a thermal conductivity detector (TCD), which reacts not only to water but also to all compounds except the carrier gas [45].

However, it should be noted that all of the aforementioned methods have disadvantages. The most critical issues affecting their efficient implementation within TMD units include the need for sample collection, the use of special reagents and analytical equipment, and high equipment and safety requirements. As a result, these methods have primarily found application in laboratory settings. The methods discussed are not intended for use with hot or boiling liquids, for real-time monitoring of the dynamics of the thermomechanical dehydration process. Therefore, there is an urgent need to conduct additional research in this area and establish the patterns of boiling of the aqueous phase from water-hydrocarbon emulsions.

2. Experimental

For the development of a method for continuous monitoring of water content during thermomechanical dehydration, samples were selected from highly stable water-hydrocarbon emulsions of natural and technogenic origins across various industries. The selected samples included sludges of cutting fluids (samples 1–4), an intermediate layer from oil and gas production department (sample 5), oil sludge from field facilities (sample 6), a mixture of oil waste after steam cleaning of railway tanks (sample 7), and a high-viscosity emulsion (sample 8), and a mixture of liquid pyrolysis products (sample 9). The characteristics of the samples are presented in Table 1. Using the mathematical modeling method described in Ref. [8] and based on the physicochemical properties of the raw materials, a preliminary assessment of the thermal stability of the emulsions was conducted. Emulsions with high retention capacity for water globules (samples 7–9) and medium retention capacity (samples 1–6) were identified.

The experimental setup is shown in Fig. 1. The diameter of the thermally insulated container of the experimental TMD installation is 0.16 m, and its height is 0.4 m. To control the temperature of the thermally insulated container, thermocouples (labeled as 11) are installed in the thermocouple pipes at both the upper and lower parts of the TMD unit. Additionally, a mixing device shaft is located within the pipe (not shown in Fig. 1). At the bottom of the heat-insulated container, there is drain pipe (labeled as 6) equipped with a drain valve for product discharge. A frame mixer driven by an electric motor serves as the mixing device.

A sample of water-hydrocarbon emulsion is loaded into a heat-insulated container 4. The filling level of the device is set at 60%. The

Table 1
Characteristics of water-hydrocarbon emulsions

Sample	Feedstock	Water/% (mass)	Hydro-carbons/% (mass)	Mechanical impurities/%	Kinematic viscosity at 20 °C, /mm^2·S^{-1}	Relative density/ g·cm^{-3}	Pour point/°C
1	Cutting fluid decomposition sludge	36.8	59.7	3.5	269	0.925	−15
2	Total sludge of cutting fluids	31.9	67.6	0.5	272	0.910	−12
3	Cutting fluid purification sludge, settling stage	46.4	51.6	2	464	0.950	−10
4	Cutting fluid purification sludge, flotation stage	34.6	53.4	12	276	0.920	−20
5	Intermediate layer from oil and gas production department	52.8	45.7	1.5	219	0.895	−10
6	Oil sludge from field facilities	36.2	63.4	0.4	260.4	0.925	−10
7	Mixture of oil waste after steam cleaning of railway tank	14.98	75.5	9.52	106.3	0.900	−7
8	High viscosity water-in-oil emulsion	31.26	68.2	0.54	254.91	0.921	−12
9	Mixture of liquid pyrolysis products	26.31	72.5	1.19	318.22	0.920	−15

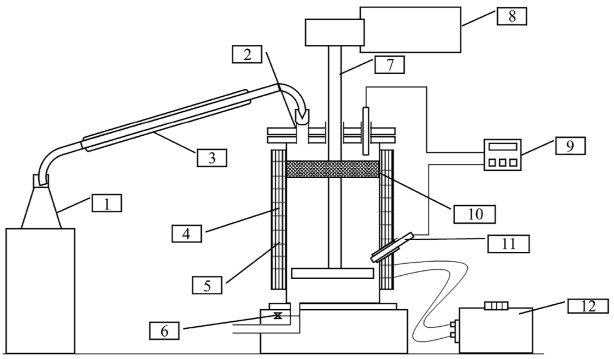

Fig. 1. Experimental unit for thermomechanical dehydration. 1—distillate receiver flask; 2—cover of a thermally insulated container; 3—condenser-refrigerator; 4—thermally insulated container; 5—heating element of a thermally insulated container; 6—pipe for draining the bottom product with a drain valve; 7—mixing device; 8—drive mixing device; 9—computing unit with indication of temperatures (controller), dT/dt coefficient; 10—layer of nozzles-droplet separator; 11—temperature sensors (thermocouples); 12—heating power regulator.

container is closed with a lid 2, and a condenser-refrigerator 3 is connected to a distillate receiver flask 1 through a pipe. Heating is applied through the wall of the container 4, while constant mixing of the liquid is maintained by a stirring device 7 to avoid overheating (150 r·min^{-1}) and facilitate liquid transfer. The resulting vapors (water and light hydrocarbons) are condensed in the condenser-refrigerator 3 and discharged into the distillate receiver flask 1. Depending on the raw material, the process duration ranges from 50 to 90 min. The distillate is subsequently separated in a separatory funnel into a hydrocarbon layer and an aqueous layer.

During the process, the temperature of the boiling liquid is recorded at regular intervals, and samples of the bottom liquid are taken, and the water content in the samples is determined by any method. In this particular case, the accuracy of the thermomechanical dehydration method is determined by the Dean-Stark method (ASTM D95—13(2018)). In the computing unit, the change in the temperature of the boiling liquid over time is recorded, and the rate of temperature increase, denoted as dT/dt, is calculated. This represents the coefficient of the temperature change rate of the boiling emulsion over time dT/dt, which is the derivative of temperature with respect to time.

3. Results and Discussion

Thermomechanical dehydration of samples 1–9 is conducted. A visual inspection of the heating surface shows that after the entire testing cycle, there are no polymerization or condensation products present. Initially, the process of water evaporation as a function of temperature is studied (Figs. 2–4). To provide a clearer understanding of the material presented in the article, the graphs display the results of dehydration for no more than three samples.

As can be seen from the graphs, more than 50% of the water evaporates when the raw material is heated to a temperature of 105 °C. The water content drops to 5% when the dehydration temperature reaches 110 °C. Fig. 2 shows graphs of the dependence of the residual water content on the boiling temperature of the emulsion for samples 1–3. Notably, when the temperature reaches the range of 140–150 °C, complete dehydration of the boiling emulsion occurs. Therefore, the possibility of continuously determining the content of residual water and the moment of completion of dehydration of the boiling emulsion based on its temperature is shown. For samples 4–6, the final dehydration temperature falls within the range of 130–150 °C (Fig. 3), while for samples 7–9, which represent highly stable emulsions, the final dehydration temperature is in the range of 150–170 °C (Fig. 4).

The second parameter is based on the study of the dependence of the residual water content on the rate of temperature increase dT/dt at a constant thermal heating power (3 kW). The change in dT/dt derivative is to be determined from the moment the boiling point is reached or the heating of the dehydrated product begins (Figs. 5–7).

Boiling of the emulsion and evaporation of the aqueous phase at high water content are accompanied by a minimal increase in temperature per unit time within the apparatus, resulting in a slight change in dT/dt. As the water content decreases during the evaporation process, less heat is required for the evaporation of water and other volatile components. This reduction in heat consumption leads to a significant acceleration in the growth of the

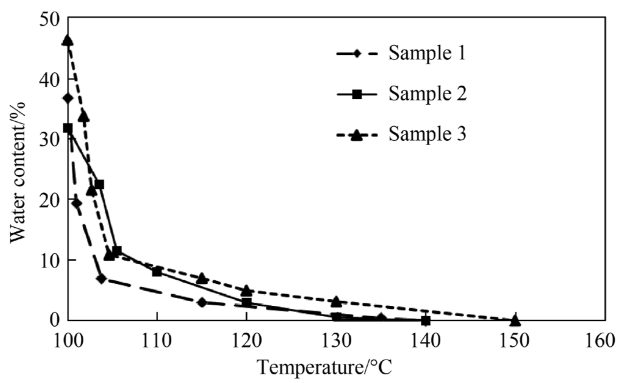

Fig. 2. Dynamics of dehydration of samples 1–3.

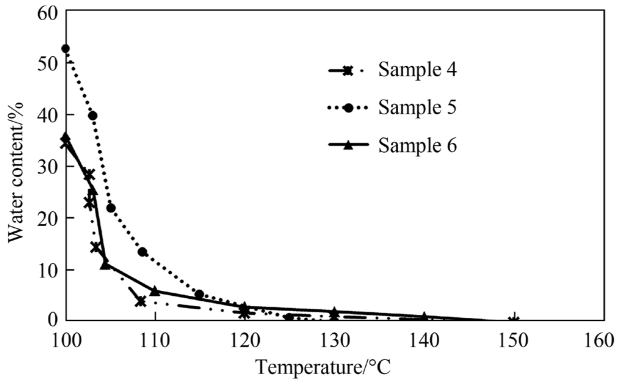

Fig. 3. Dynamics of dehydration of samples 4–6.

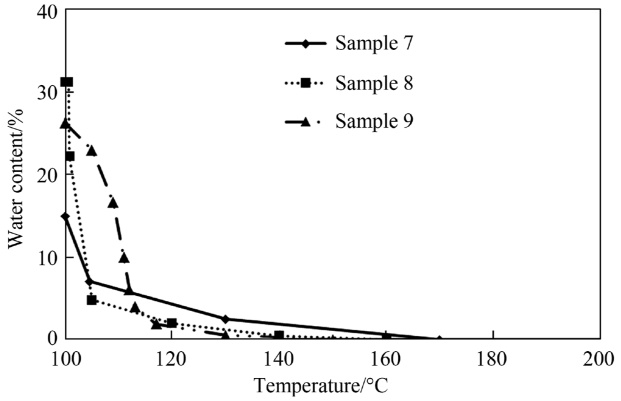

Fig. 4. Dynamics of dehydration of samples 7–9.

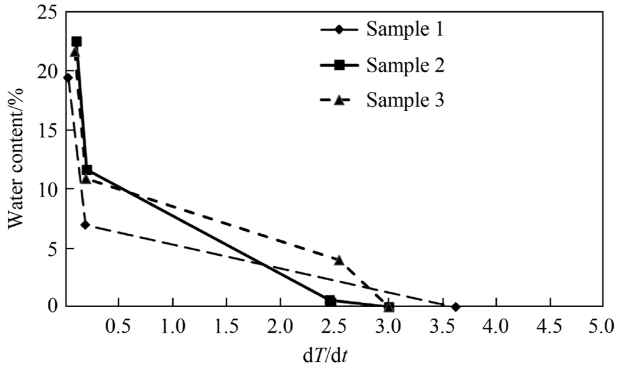

Fig. 5. Dependence of water content in emulsions as a function of d*T*/d*t* for samples 1–3.

process temperature, thereby enhancing the rate of temperature increase.

Figs. 5–7 demonstrate that for samples 1–3, complete dehydration of the boiling emulsion occurs when d*T*/d*t* reaches the value of 3.0–3.6. For samples 4–6, this occurs in the range of 3.0–4.6, while for samples 7–9, it occurs in the range of 3.5–4.0. Therefore, based on d*T*/d*t*, it is possible to continuously monitor the residual water content in products and determine the moment of complete dehydration of the boiling emulsion.

Achieving d*T*/d*t* values between 3.0 and 5.5 or higher indicates the complete dehydration of the boiling emulsion. Therefore, the time interval (moment) marking the end of dehydration can be

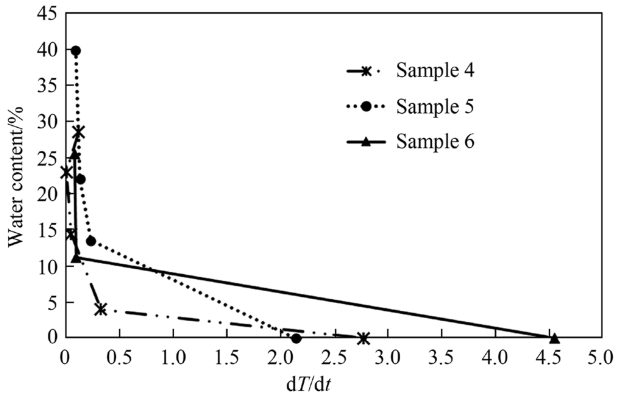

Fig. 6. Dependence of water content in emulsions as a function of d*T*/d*t* for samples 4–6.

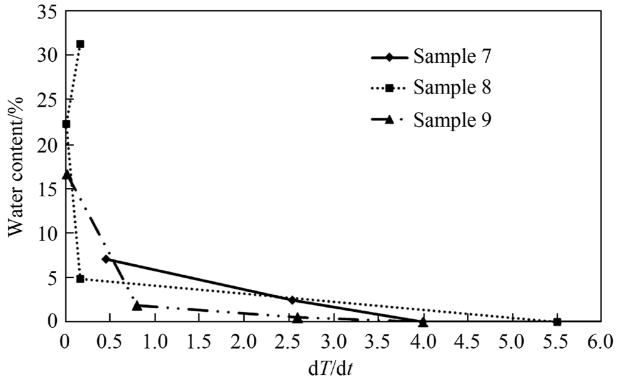

Fig. 7. Dependence of water content in emulsions as a function of d*T*/d*t* for samples 7–9.

established. The combined use of these two criteria allows for increased the accuracy in determining the current water content in a boiling emulsion and facilitates the identification of the precise moment of complete dehydration of the emulsion in real time. This methodology can be applied to a wide range of emulsions of various compositions. Thus, monitoring and controlling the thermomechanical dehydration process can be accomplished using basic, simple devices that consist of temperature sensors (such as thermoelectric converters or thermal resistor) and a computing unit (computers, industrial controllers). These devices provide real-time indications of temperature and the rate of temperature increase d*T*/d*t*.

4. Conclusions

A method for continuous monitoring of water content in boiling emulsions has been developed to control and automate the process of thermomechanical dehydration of highly stable water-hydrocarbon systems. The initial and end boiling points of the aqueous phase in these emulsions differ from the boiling point of water (100 °C) and can lie within a wide range. This variability presents challenges in predicting and implementing an automated thermomechanical dehydration process, which has proven effective for dehydrating highly stable emulsions that resist breakdown by traditional methods (based on the settling of the aqueous phase) and have therefore accumulated over time.

As part of the research, samples of water-hydrocarbon emulsions of different origins and covering different areas of industrial enterprises were examined. For the first time, experimental

dependences of the residual water content during the thermomechanical dehydration were constructed based on process temperature and the rate of temperature increase dT/dt. It is proposed to use these obtained dependences for continuous, real-time monitoring of the residual water content in a boiling emulsion and monitoring the moment of its complete dehydration. This approach can be applied to various processes that involve evaporating water from emulsions.

Research has shown that achieving a boiling emulsion temperature of 130–170 °C or higher, and/or achieving dT/dt values between 3.0 and 5.5 or above, indicates complete dehydration of the boiling emulsion. Furthermore, this method is characterized by its simplicity and cost-effectiveness, requiring only temperature sensors and a computing unit. Importantly, it does not necessitate constant sampling and can be implemented in any industrial or laboratory apparatus for thermomechanical dehydration without significant capital costs or operational restrictions.

CRediT Authorship Contribution Statement

A. Safiulina: Conceptualization, Writing – original draft, Writing – review & editing. S. Khusnutdinov: Investigation. I. Khusnutdinov: Conceptualization, Project administration, Supervision. I. Goncharova: Investigation.

Declaration of Competing Interest

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

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