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INVESTIGATION OF CHARGE AND DISCHARGE REGIMES OF NANOMODIFIED HEAT-ACCUMULATING MATERIALS

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Досліджені режими заряду/розряду наномодифікованих парафінів. Вивчені наступні теплофізичні параметри: теплопровідність та теплоємність. В режимах заряду/розряду проявляється ефект «стежачого теплового контакту». Теплопровідність збільшується до 0,48, 0,42 та 0,36 Вт/м°С відповідно УНМ-МД, УНМ-М, УНМ, відносно вихідної теплопровідності – 0,25 Вт/м°С. Змінюється екстремум на графіку температурної залежності теплоємності (57, 63 и 72 °С, відповідно для УНМ, УНМ-М та УНМ-МД)

Ключові слова: тепловий акумулятор, парафін, модифікація, вуглецеві нанотрубки, теплопровідність, теплоємність, заряд/розряд

Исследованы режимы заряда/разряда и теплофизические параметры (теплопроводность и теплоемкость) наномодифицированных парафинов. В режимах заряд/разряд проявляется эффект «следящего теплового контакта». Теплопроводность увеличивается до 0,48, 0,42 и 0,36 Вт/м°С в случае УНМ-МД, УНМ-М, УНМ, относительно исходной теплопроводности – 0,25 Вт/м°С. Изменяется экстремум на графике температурной зависимости теплоемкости (57, 63 и 72 °С, соответственно для УНМ, УНМ-М и УНМ-МД)

Ключевые слова: тепловой аккумулятор, парафин, модификация, углеродные нанотрубки, теплопроводность, теплоемкость, заряд/разряд

1. Introduction

The efficiency increase of majority of technological processes in the chemical and food industries, and also in other fields, using thermal apparatuses, is tied with conservation of thermal energy. The thermal energy can be conserved by using heat accumulators. Heat accumulators are based on heat-accumulating materials, which can with or without a phase change [1, 2]. Phase-transition heat-accumulating materials allow reducing the mass and size of heat accumulators at least 2 times, and with optimization and effective placement of heat exchangers – 2.5 times. There are strict requirements for heat-accumulating materials. These requirements are related to stability during operation, absence of toxic components, fire and explosion safety. The economic aspect of using heat accumulators should also be noted. The main objective of heat accumulators is to improve the energy efficiency of equipment, and in this case, the cost must correspond to economic effect, i.e. the heat-accumulating materials should be available and affordable. In that regard, sodium acetate, sodium sulfate or hydrocarbons, such as paraffins, can be used. First two materials require special equipment; that is resistant to corrosion and have a limited number of charge-discharge cycles. The paraffin hydrocarbons have longer cycle life and are available in sufficient amounts, as in some cases, they are a by-product of oil processing. Another advantage is that phase-change temperature can be set by choosing an appropriate paraffin from C_xH_y group. The disadvantage of using paraffins for heat accumulation is their low thermal conductivity, which results in a low rate of charge/discharge.

2. Literature review and problem statement

Paraffin modification with carbon nanomaterials (possessing high thermal conductivity) is one of the most effective methods of improving thermophysical parameters. The higher the content and thermal conductivity of the introduced material, the higher the effect on the thermophysical properties of the matrix. From a variety of materials that can affect thermal conductivity of heat-accumulating materials, it is worth outlining directions with utilizing metals, carbon materials (graphite, graphene, carbon nanotubes) and secondary materials (clay). The influence of silver nanoparticles on thermophysical parameters of phase-transition material has been studied in the paper [3]. The other research [4] has conducted an analysis of copper nanoparticle influence on the heat-accumulating composite. However, the distribution of metal nanoparticles in the volume of phase-transition material is poor, which leads to use of metal foam [5]. Use of metallic materials leads to the formation of unstable composites that shear during charge/discharge. This leads to the emergence of a new direction that deals with carbon nanomaterials (carbon nanotubes) [6, 7]. The influence of nanotube diameter on thermophysical properties of paraffin has been experimentally demonstrated in the paper [8]. The authors of the paper [9] have proposed to use montmorillonite, which is a clay mineral from phyllosilicate group, as an additional component. The paper [10] studies the influence of graphite on thermophysical parameters of eutectic mixtures. The research on the influence of graphene on thermophysical properties of paraffins has been conducted in the paper [11]. Aside from experimental studies, computer modeling of graphene [14], specific surface area in particular, has found extensive application. The presented approach is valuable when choosing required parameters for material modification. The paper [15] studies paraffin microencapsulation using silica.

Use of metal materials as additives increases the thermal conductivity of paraffins several times, but the reduction of specific heat capacity, proportional to the amount of introduced thermal conductor is observed. Modification of paraffin with metal particles and foams also leads to decrease of the energy density of heat accumulators [10].

A number of researchers, relying on high thermophysical properties of carbon nanomaterials, have used in their works carbons nanotubes, [6, 7], mixtures of carbon nanotubes and montmorillonite, [9], graphene [11, 12]. Thermal conductivity of some carbon nanotubes can be above 3000 W/mK [16], and for diffident types of graphene it is in the range of 500 to 5000 W/mK [11]. Upon addition of 0.4 % by volume of oriented multi-walled carbon nanotubes to s160 and s760 elastomers [18], the thermal conductivity of the composition has increased 2 and 4 times correspondingly. A similar result was obtained in the paper [19]. Upon addition of 4 % by weight of non-oriented MWCNT to silicone elastomer Sylgand 160 (USA), the thermal conductivity has increased almost 2 times.

However, upon modification of paraffins with carbon nanotubes, the thermal conductivity has increased by no more than 30 % from the initial value [17]. Additionally, the concentrations of carbon nanotubes were several times higher than those used in [17, 18]. The hidden potential of using highly thermally-conductive carbon nanomaterials is not realized, mainly because of intra-phase phenomena, which interfere with the propagation of phonons from the volumetric material into a nanoparticle and between individual contacting nano-sized components. The direction of future research reflecting the modern state of the problem of increasing the thermal conductivity of paraffins modified with carbon nanomaterials, has split into two directions.

The first direction is aimed at creation of multi-component systems, that, aside from carbon nanotubes, include an additional material that improves thermal conductivity and provides a better heat transfer between phases.

Intercalation of paraffin into interlayer spaces of montmorillonite has led to the 38 % increase of thermal conductivity and provided thermal stability of the composition for 100 charge-discharge cycles [9]. As a result of combined use of montmorillonite and carbon nanotubes as modification components, the 73 % increase in thermal conductivity of the initial paraffin matrix has been achieved [9].

The main idea of the second direction is control of physical mechanisms that limit heat transfer through carbon nanomaterials, i. e. components and inter-phase boundaries of the composition. The thermal conductivity mechanisms are defined by the linear size, defectiveness, number of atomic layers and concentration of carbon nanotubes in the paraffin matrix. The paper [8] experimentally demonstrated that the contact area between the nanotubes distributed in paraffin depends on the diameter of nanotubes. The experimental result obtained is in good agreement with the results of theoretic work [19]. In the paper [8], it has also been established, that thermal conductivity between contacting carbon nanotubes can increase up to 27 times, with an increase of diameter by 58 nm. When investigating the compositions containing carbon fibers, the authors of the paper [6] claim that the decrease of contacts between highly thermally conductive particles should be minimized. In other words, the longer the fiber, the better it is involved in heat transfer. Modern researches demonstrate an increase of the suspensions' thermal conductivity with increasing length of graphene scales [19]. Such effect is explained by the ballistic regime of low-frequency acoustic phonon thermal conductivity [11]. Also, the paper [11] presents experimental results, that demonstrate an increase of the paraffin's thermal conductivity by almost 2 orders when graphene is used. The concentration of graphene in paraffin was 20 % wt., scale thickness and length were 1–8 nm and 500–10000 nm correspondingly [11]. There are a few possible reasons for such significant increase of thermal conductivity. First, the distribution of graphene scales that create a "net" that provides high heat transfer inside the nanocomposite and strong binding of graphene flakes with hydrocarbon-based matrix [11]. The strong dependency of thermal conductivity on the graphene content in paraffin should also be noted. When the authors of the paper [11] have used concentration 10 times lower than in [13], the thermal conductivity of the nanocomposite has increased by only 60 %.

Thus, the boundary region between individual materials in the composite and geometric parameters of carbon nanomaterials have a significant influence on volumetric thermal conductivity.

In cases when paraffins are used for solar energy systems, it is not always critical, because processes are not limited in time. In order to solve all mentioned problems, various approaches that are based on the use of various materials and technical methods, have been applied. The silica was used in the paper [15], and the authors of papers

[5, 6] propose to modify paraffin using carbon nanotubes. The use of additional additives leads to decrease of heat capacity and complication of production technology. In addition, the solution methods that could be effective for rapid thermal processes that occur during exothermic chemical reactions have not been formed. Thus, the question related to the improvement of thermal parameters of paraffins that can be used for heat accumulators remains open. The method of improving thermal parameters of paraffins that is based on modification using carbon nanotubes could be especially effective. For modified paraffins, it is necessary to study the thermophysical parameters and to evaluate the charge-discharge process.

3. The aim and objectives of the study

The aim of the presented work is to study the charge and discharge processes of thermal energy in nanomodified materials with the controlled phase transition.

In order to achieve the set aim, the following objectives must be fulfilled:

– to study the influence of carbon nanomaterials of the “Taunit” series CNM, CNM-M and CNM-MD on thermophysical parameters 0 of paraffins;

– to study the effects occurring during different charge-discharge regimes;

– to study the charge-discharge time of nanomodified materials.

4. Synthesis and analysis methods of nanomodified paraffin

4. 1. Synthesis of nanomodified paraffin

In order to investigate the influence of different CNM types on thermophysical parameters of heat-accumulating materials, 3 nanomodified paraffin samples are studied. The difference lies in different CNM (carbon nanomaterial of the “Taunit” series): CNM, CNM M and CNM MD, according to the classification of PLC “NanoTechCent” Tambov, Russia. CNM of the “Taunit” series is nano-scale carbon fiber-like formations with an inner channel (Fig. 1). According to the classification, CNM of the “Taunit” series~~;~~ are ~~a~~ characterized as multi-walled carbon nanotubes with the “lampshade” structure. The characteristic sizes of MWCNT are in the range: outer diameters D_{out}=15...40 nm; inner – D_{in}=3...6 nm.

Microphotographs of CNM presented in Fig. 1 demonstrate morphological differences of materials.

4. 2. Modification of paraffin

An amount of CNM (different type of CNM for each sample) was placed into a 0.002 m^3 silicone container, then the P2 paraffin heated to 80 °C was added. The mixture was ultrasonically treated at 22.5 kHz for 1.5 h and mechanically stirred (100 rpm) at a temperature of 70 °C. As a result, the black non-transparent material was obtained. The content of CNM in paraffin was 20 % wt., for CNM-M – 2 %, and for CNM-MD – 1.5 %. The prepared materials were tested in various engineering applications, among which are regenerative heat exchangers for personal respirators [21] and industrial heat accumulators [22].

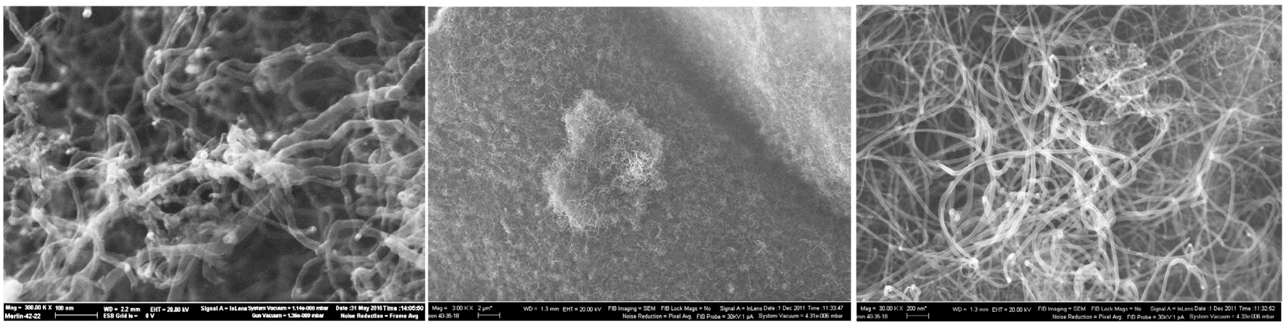
4. 3. Methods for analysis of nanomodified paraffin

In order to study the distribution of CNM in paraffin, the analysis using the Micromed-1 optical microscope (Russian Federation) was carried out.

Measurement of thermophysical parameters of modified paraffin was carried out using apparatuses IT – λ – 400 (measurement of thermal conductivity (W/m°C) Fig. 2, 3) and IT – S – 400 (measurement of heat capacity, (J/kg°C)) in a monotone heating mode at an average rate of 0.2 °C/s under adiabatic conditions.

Fig. 2. Automated measurement system IT

In order to study charge-discharge regimes, a device with the controlled heat flow source was used. The device (Fig. 4) is equipped with 5 sections, in order to compare the charge parameter of heat-accumulating materials. Through all the sections, which are 1cm steel tubes, water flows with a temperature in the range of 20 to 95 °C at a flow rate of 0.1 to 5 m^3/h. This provides the specific power of released heat from 20 to 300 W/m. Metallic tubes are located in thermal-insulating layers of styrofoam.



a *b* *c*

Fig. 1. Microphotographs of the “Taunit” series CNM, scale 200 nm: *a* – CNM; *b* – CNM-M; *c* – CNM-MD

Fig. 3. IT measuring cuvette

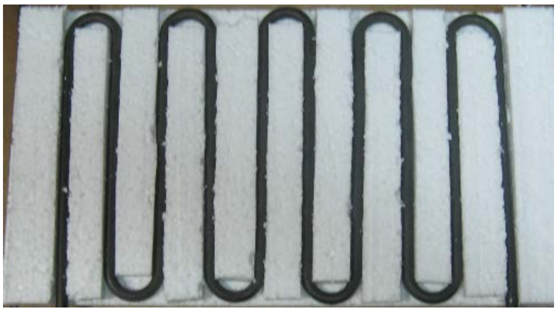


Fig. 4. The device for studying charge-discharge regimes of paraffin modified with carbon nanotubes

The paraffin, modified with carbon nanotubes is placed on top of heat exchange sections with a layer thickness of 5mm. During the investigation, the non-contact method that is realized in the Testo 871-1 thermal imager (Germany) was used.

5. Results of the nanomodified paraffin sample analysis

In order to evaluate the effectiveness of CNM distribution in paraffin, the analysis using the Micromed-1 optical microscope (Russia) (Fig. 5–7) has been carried out.

Fig. 5 shows the distribution of CNM-M in paraffin. The distribution is disordered with pronounced empty zones.

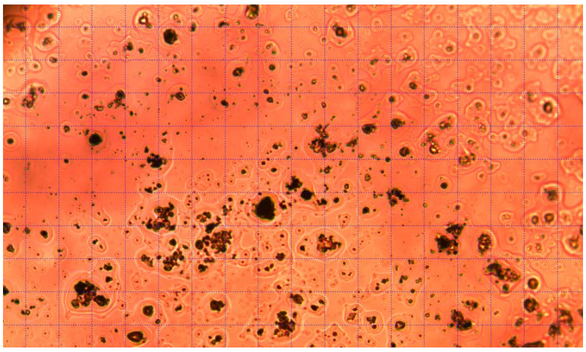


Fig. 5. Distribution of CNM agglomerates in paraffin

Fig. 6 shows the distribution of CNM in paraffin. The distribution is disordered with pronounced empty zones, but unlike CNM-M there are smaller agglomerates and the area of empty zones is smaller.

Fig. 7 shows the distribution of CNM-MD in paraffin. The distribution is disordered with pronounced empty zones, but unlike CNM there are larger agglomerates.

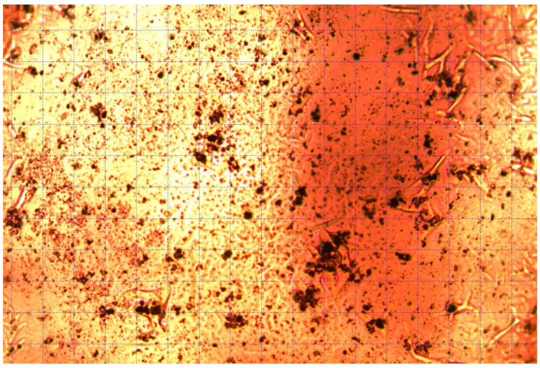


Fig. 6. Distribution of CNM agglomerates in paraffin

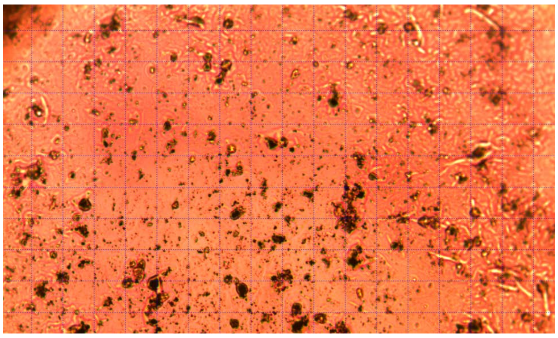


Fig. 7. Distribution of CNM-MD agglomerates in paraffin

According to the experimental studies (Fig. 8), the temperature dependencies of pure paraffin and nanomodified paraffins with different types of CNM, CNM-M and CNM-MD (Fig. 8) have been established.

Fig. 9 shows the studies of the temperature dependency of thermal conductivity of paraffin modified with carbon nanotubes.

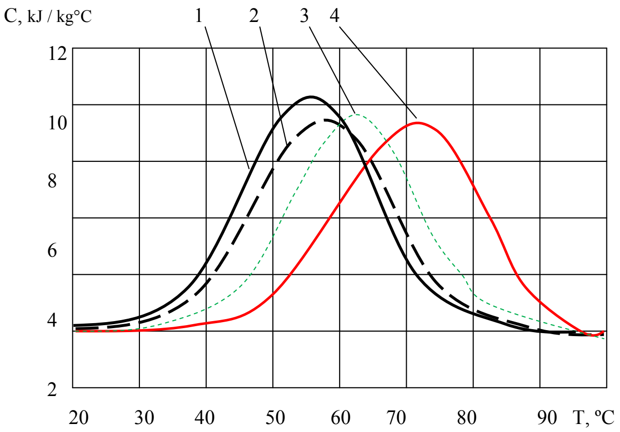


Fig. 8. Temperature dependency of paraffin modified with carbon nanotubes: 1 – paraffin; 2 – paraffin modified with CNM; 3 – paraffin modified with CNM-M; 4 – paraffin modified with CNM-MD

Fig. 10 shows the distribution of temperature field in the device sections in the regime before the phase transition of the heat-accumulating material. This regime is the least effective during charge-discharge, because in the temperature range of 20–40 °C heat exchange intensity is low and under conditions of low thermal conductivity leads to incomplete discharge.

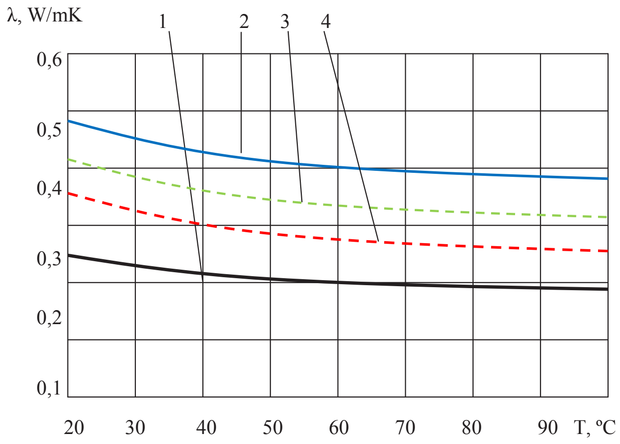


Fig. 9. Temperature dependency of paraffin modified with carbon nanotubes: 1 — paraffin; 2 — paraffin modified with CNM; 3 — paraffin modified with CNM-M; 4 — paraffin modified with CNM-MD

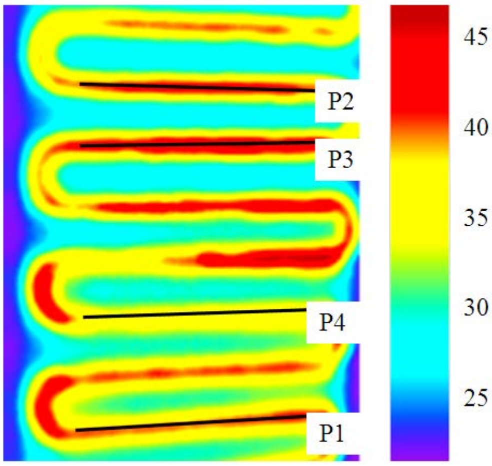


Fig. 10. Thermogram: P1 — CNM, P2 — CNM-M, P3 — CNM-MD, P4 — pure paraffin

Fig. 11 shows the thermal profile in the sections corresponding to the samples P1 – CNM, P2 – CNM-M, P3 – CNM-MD, P4 – pure paraffin.

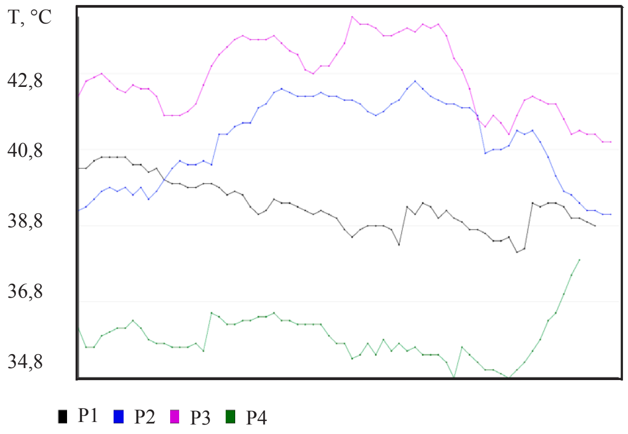


Fig. 11. Thermal profile along the thermogram lines: P1 — CNM, P2 — CNM-M, P3 — CNM-MD, P4 — pure paraffin

Fig. 12 shows the study of charge and discharge times of nanomodified paraffin.

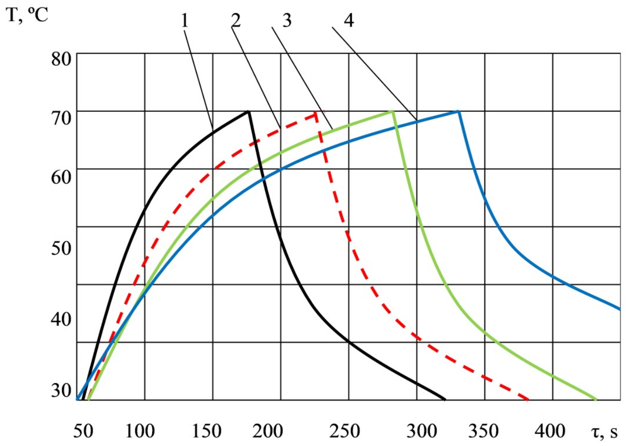


Fig. 12. Charge and discharge times of paraffin and modified paraffins: 1 — paraffin modified with CNM-MD; 2 — paraffin modified with CNM-M; 3 — paraffin modified with CNM; 4 — paraffin

Table 1 shows residual heat after discharge.

Table 1

Values of residual heat in heat-accumulating material

№	Sample № 1	Sample № 2	Sample № 3	Sample № 4
Q_{res}, (J)	280.4	164.6	145.8	112.3

Data presented in Table 1 shows that paraffin modified with CNM-MD has the least amount of residual heat. For pure paraffin (sample № 1), the highest value of residual heat is observed, which is related to the low value of thermal conductivity. Samples № 2 (CNM) and № 3 (CNM-M) have similar values of residual heat.

6. Discussion of results of studying the characteristics of nanomodified paraffin samples

When studying the structure of nanomodified paraffin using an optical microscope, the CNM agglomerates of various sizes (Fig. 5, 6) are observed, with distribution being chaotic in character. Upon visual evaluation, the best distribution is characteristic for CNM-modified paraffin. At the same time, the formation of large agglomerations with a large distance between individual agglomerates is characteristic for paraffin modified with CNM-M. Based on these images, it can be concluded that modification is local in character, in which the modified part of paraffin is located in paraffin that is in the initial state.

According to the experimental research (Fig. 8), the temperature dependencies of heat capacity for paraffins modified with CNM, CNM-M and CNM-MD have been found. The temperature dependency of nanomodified paraffin shows that with the use of different types of carbon nanomaterials, a shift of the graph extremes occurs. A change of the phase-transition temperature is observed: 57, 63 and 72 °C for CNM, CNM-M and CNM-MD correspondingly. The highest shift is observed for modification with CNM-MD.

By comparing the temperature dependencies, it can be concluded that modification of paraffin with different types of CNM allows changing the thermophysical parameters of paraffin. This leads to a change of the phase-transition temperature and broadening of phase-transition boundaries. This allows increasing the effectiveness of the heat-accumulation system, because of phase-transition range broadening, which in turn, leads to an increase of heat capacity by 30–50 %.

Paraffin modification with carbon nanotubes allows controlling the phase-transition parameters, which provides acquisition of various types of temperature dependencies of heat capacity, thermal conductivity and physical-mechanical properties by combining various ratios of the "Taunit" series carbon nanotubes, and physical interactions in order to optimize the operation of heat accumulations under different temperature regimes.

By analyzing the results presented in Fig. 9, it follows that the best result corresponds to the sample modified with CNM-MD. At 20 °C its thermal conductivity increases from 0.25 to 0.48 W/mK. For the sample modified with CNM, the thermal conductivity increases to 0.36 W/mK, and for CNM-M – to 0.42 W/mK. With increasing temperature, the thermal conductivity decreases, which is related to the disconnection of agglomerated chains that affect heat transfer.

Fig. 10 presents a comparative distribution of the temperature field, upon analysis of which it is possible to evaluate charge efficiency under the same heat exchange conditions.

Fig. 11 presents information supplementary to Fig. 10. It can be concluded that the sample distributed in section P3 (CNM-DM) provided the fastest establishment of the maximum temperature regime, owing to the highest thermal conductivity. At the same time, the pure paraffin located in section P4 has the lowest temperature for the same time period, which is related to its lower thermal conductivity.

The best result (Fig. 12) during charge-discharge is demonstrated by sample 4 (paraffin modified with CNM-MD), which is related to higher thermal conductivity and presence of tracking thermal contact, which is more pronounced for CNM-MD.

Heat values listed in Table 1 are the values of residual heat after discharge of heat-accumulating material in heat accumulator. The value of residual heat is primarily affected by features of heat accumulator, however, under all other conditions being equal, parameters of heat-accumulating material also matter. It has been found that the least amount of residual heat corresponds to the sample with CNM-MD (Sample № 4). This is related to the effect of tracking thermal contact that is formed between the heat-accumulating material and heat-exchanger of the heat accumulator.

Tracking thermal contact is caused by volume change at the proportional change of temperature difference. Volume change of paraffin (Sample № 1) is caused by incorrect material distribution in a closed volume ("funnel" effect). This leads to the formation of crevices and shift of the heat-accumulating material inside the heat accumulator.

7. Conclusions

1. During the experimental research, the influence of CNM, CNM-MD and CNM-MD on thermophysical properties of paraffins has been found. The content of CNM in paraffins was 20 % by wt., for CNM-M – 2 %, and for CNM-MD – 1.5 %. The thermal conductivity changes with modification and the best result corresponds to the sample prepared using CNM-MD. At 20 °C thermal conductivity increases from 0.25 to 0.48 W/mK. For the sample modified with CNM, the thermal conductivity increases to 0.36 W/mK, and for CNM-M – to 0.42 W/mK. The extreme on the temperature dependency graph shifts to higher temperature values (57, 63 and 72 °C, for CNM, CNM-M and CNM-MD correspondingly).
2. The effects of tracking thermal contact that provides a more complete discharge of thermal energy have been found.
3. Nanomodified paraffins possess a lower charge time (175, 225, 280 s for CNM, CNM-M and CNM-MD correspondingly) and discharge time. This makes nanomodified paraffins more effective heat-accumulating materials than pure paraffins (charge time 380 s).

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