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Integrated Activation Techniques for Enhanced Thermal Dissolution of Barzas Coal into Liquid Hydrocarbons

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Keywords:
Thermal dissolution; Coal liquefaction; Mechanochemical activation; Ozone treatment; Ultrasonic processing; Tetralin solvent; Liquid hydrocarbons; Conversion efficiency.

Highlights:

- *Ultrasonic pretreatment increased coal conversion to 88.3%, exceeding that of conventional methods.*
- *Liquid products exhibited a high calorific value of 41.2 MJ/kg, confirming their suitability as fuel components.*
- *Combined mechanochemical and ozone activation enhanced the yield of light hydrocarbons to 58.6% of dry coal mass.*

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Abstract: The study investigates the thermal dissolution of Barzas coal using mechanochemical activation, ozone treatment, and ultrasonic processing to enhance the yield and quality of liquid products. The experimental work comprised preparing coal samples by high-energy ball milling, followed by ozone oxidation and ultrasonic pretreatment, and thermal dissolution in tetralin at 375-425 °c. The results showed that the integrated activation approach significantly increased organic matter conversion, reaching up to 88.3% at 400 °C with ultrasonic treatment, compared with 64.9% without activation. The yield of liquid fractions was 57.9% of the dry coal mass, and the light fractions exhibited a high calorific value of up to 41.2 MJ/kg, with aromatic hydrocarbon content exceeding 65%. Lower heating rates and controlled temperature conditions were essential to maximise conversion and minimise heavy residue formation. The findings highlight the effectiveness of combining mechanical, oxidative, and ultrasonic activation to improve coal dissolution and produce liquid hydrocarbons suitable for fuel components and chemical feedstock applications.

1.INTRODUCTION

In the modern world, amid growing energy challenges, coal remains a key strategic resource, providing approximately 37% of global electricity and supporting about 70% of steel production. According to estimates by the International Energy Council, fossil coal consumption will increase by another 1 billion tonnes during the first three decades of the 21st century, driven by demographic growth and the increasing industrialisation of developing countries. Global coal reserves are estimated to be sufficient to meet growing energy demand over the next several hundred years, underscoring coal's continued role in the global energy balance. At the same time, the widespread use of coal entails high environmental costs, including emissions of sulphur and nitrogen oxides and carbon, necessitating the search for new solutions for its processing and utilisation. One traditional way of using coal is direct combustion in furnaces, but this method is characterised by poor environmental safety and relatively low efficiency [1-2]. Technological approaches aimed at the complex processing of coal include gasification and hydrogenation, which occupy a special place. Coal gasification enables the production of synthetic gas and chemical products, and its development is evidenced by more than 63 gasification projects worldwide, of which 55 are intended for electricity generation and only eight focus on producing chemical raw materials. Such an imbalance emphasises that chemical processing remains in the minority. Coal hydrogenation for the production of synthetic liquid fuel is considered a technologically promising direction, as evidenced by the operation of more than 80 pilot plants in different countries. However, hydrogenation processes are still imperfect, and the resulting fuel cannot compete in cost with petroleum products. In addition, direct liquefaction of coal is complicated by the need for further processing of liquid products, which requires significant investment and the availability of specialised industrial complexes. There is another, more straightforward approach to transitioning to the production of coal-water fuel suspensions. Technologies for their preparation and transportation have been fully developed, making this route attractive for organising energy supply in areas remote from main fuel networks [3-5]. However, the focus here remains on energy generation rather than on the production of valuable chemical compounds. The coke industry, as a traditional coal consumer, has progressed and made considerable technological advances. High-performance complexes for processing coking gas have advanced significantly. At the same time, further has been slow. In addition, modern facilities for the production of chemical products often fail to meet environmental and industrial safety requirements, and their reconstruction requires significant investment. These circumstances, along with tightening environmental standards and the need to improve resource efficiency, underscore the importance of seeking alternative coal-processing methods [6-9]. Feedstock for the chemical industry. Direct use of coal in the chemical industry can provide an economically efficient and environmentally sound alternative to petrochemical production. However, to achieve this goal, it is necessary to advance and test non-fuel applications of coal, including the production of high-value organic compounds. One promising strategy is the thermal dissolution of coal to convert its organic matter into liquid products of specified quality. Previous studies at the Institute of Coal and Coal Chemistry of the Siberian Branch of the Russian Academy of Sciences showed that when Barzas coals are dissolved in tetralin, the conversion of organic matter in the 375–425 °C temperature range reaches 75–85%. The use of preliminary mechanochemical activation and ozonolysis further intensifies the process at low temperatures, improving the yield of liquid fractions and altering their composition. For example, by varying the temperature and heating rate, it is possible to obtain both medium- and light-hydrocarbon fractions, as well as organic binders for coal briquette production and road construction [10,11]. However, these data remain laboratory-based, and several engineering and economic barriers must be overcome before practical implementation of the technology. The relevance of the authors' work lies in the fact that, despite the long-term development of coal chemistry technologies, Russia still lacks industrial-scale development of complex coal processing aimed at producing chemical products rather than energy or fuel. The existing structure of coal use is mainly focused on combustion and, to a lesser extent, on coking [12-14]. Meanwhile, modern trends towards greater resource efficiency and lower atmospheric emissions necessitate the development of technologies for complex coal processing, including the production of targeted organic compounds and synthetic fuels. Introducing thermal dissolution processes, activation methods, and controlled processing modes can serve as an essential link in developing such technologies [15-17]. In addition, establishing the thermodynamic and kinetic laws of coal dissolution and selecting suitable solvents, catalysts, and process conditions are not only of fundamental importance but also of high practical value. The article presents a study of the parameters of coal thermal dissolution, the determination of optimal conditions to achieve maximum conversion of organic matter, and the intentional modification of liquid product composition. Research gap and novelty. Despite extensive studies on coal liquefaction and thermal dissolution, most prior approaches employ a single pretreatment (mechanochemical, oxidative or ultrasonic), which limits low-temperature reactivity and control over product distribution. The effects of an integrated sequence of mechanochemical disruption, mild ozone oxidation, and ultrasonic pre-treatment, coupled with carefully controlled heating rates in a hydrogen-donor solvent, on the conversion of organic matter at 375–425 °C, selectivity towards light fractions, and the onset of solvent thermogenic degradation remain insufficiently resolved. Here we explicitly address this gap. The novelty of this study lies in a head-to-head evaluation of a three-stage activation sequence before dissolution in tetralin, and in quantifying its combined effect on conversion (up to 88.3% at 400 °C), light-fraction yield, and product quality relative to non-activated or singly activated baselines under otherwise identical conditions. Objectives and hypotheses. We can test two hypotheses. (H1): Integrating mechanochemical, ozone, and ultrasonic steps yields complementary defect chemistry and accessibility, thereby increasing conversion and light-fraction yield relative to any single step. (H_2): Limiting the heating rate suppresses secondary condensation and solvent cracking, thereby improving selectivity and stability of light fractions. Accordingly, our objective is to define operating windows (temperature, dwell time, heating rate) that maximise conversion while minimising heavy residues in the integrated activation route.

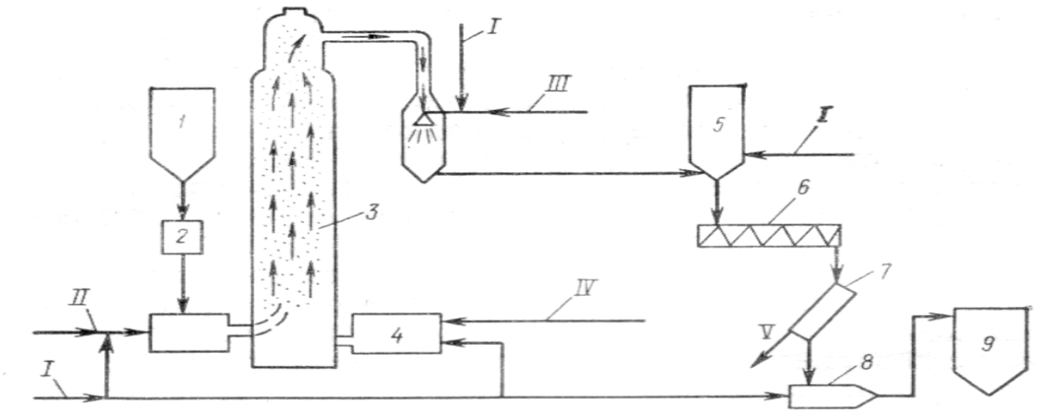

Fig. 1 The Installation for Thermal Regeneration of Highly Dispersed Activated Carbon: I–IV — Air, Steam, Water and Natural Gas Supply Pipelines; V — Waste Discharge Pipeline; 1 — Spent Coal Collector; 2 — Dispenser; 3 — Regenerator; 4 — Combustion Chamber; 5 — Separator; 6 — Auger; 7 — Filter; 8 — Pneumatic Conveyor; 9 — Regenerated Coal Collector.

2.RESEARCH METHODS

Within this study, a series of experiments was conducted to investigate the thermal dissolution of coal and to obtain liquid products with controlled composition. The experiments comprised step-by-step mechanochemical activation of the initial coal, subsequent heat treatment in a solvent environment, and product analysis. The primary focus was coal from the Barzas deposit, characterised by high vitrinite content and a metamorphic grade that provides favourable conditions for dissolution. Mechanochemical activation was performed in a Pulverisette 7 planetary ball mill (Fritsch) equipped with 10 mm tungsten balls. The process was run at 600 rpm for 45 minutes to disrupt the coal's crystallite structure and increase its reactivity. After activation, the material was immediately subjected to ozone-acid treatment in a 3-litre glass reactor equipped with a bubbler, feeding an ozone-oxygen mixture at 80 g/m^3 and 25°C for 60 minutes. This stage induced partial oxidation of the coal particle surfaces, facilitating further solvent penetration into the pores. Thermal dissolution was carried out in a Parr 4560 autoclave at pressures up to 15 MPa. Tetralin, with a purity of at least 98%, was used as a solvent. Tetralin was selected as a hydrogen-donor solvent with a high normal boiling point and a proven ability to stabilise radicals formed during bond scission in coal matrices at ≥ 350 °C. In contrast to lower-boiling protic solvents such as methanol or ethanol, tetralin maintains liquid-phase operation across the entire 375–425 °C window, facilitates hydrogen transfer to reactive fragments, and reduces retrogressive condensation. Together with controlled heating rates, these features support higher conversion and a larger share of light fractions under our conditions. The mass ratio of coal to solvent was 1:5. The experiments were stirred at 200 rpm. In selected series, the temperature was varied from 375 to 425 °C in 25 °C increments to determine the optimal dissolution mode. After the hold, the autoclave was cooled to 50 °C, the pressure was reduced to atmospheric, and the suspension was filtered using a Buchner System 3000 vacuum filter with a porous ceramic membrane. The resulting liquid fractions were fractionated by vacuum distillation at a residual pressure of 10 kPa, yielding light and medium hydrocarbon fractions and high-boiling residues. The full activation–dissolution pipeline is as follows: mechanochemical activation in a planetary mill → low-temperature ozone oxidation of the activated powder → ultrasonic pre-treatment of the slurry → thermal dissolution in tetralin at 375–425 °C under stirring with a controlled heating rate → cooling, depressurisation, and filtration → vacuum distillation into light and medium fractions, with heavy residues retained for analysis.

Fig. 2 The External View of the Parr 4560 Autoclave.

In addition, experiments were conducted to determine the rate of the organic matter conversion at different heating rates from 3 to 10°C/min and to study the effect of preliminary ultrasonic treatment, implemented using a Hielscher UP400S ultrasonic unit operating at a frequency of 24 kHz and a power of 300 W for 30 minutes. These stages provided a comprehensive understanding of the mechanisms governing the destruction and dissolution of coal under the given conditions.

3.RESULTS AND DISCUSSION

During the work, a comprehensive study was conducted, encompassing several stages of coal raw material processing and analysis of the resulting products. First, the original coal from the Barzas deposit was subjected to mechanochemical activation to enhance reactivity. For this purpose, 50 g of coal samples were loaded into the drum of a Pulverisette 7 planetary ball mill, where the activation process was carried out at 600 rpm. The duration of the mechanochemical effect was 45 minutes. The use of 10 mm-diameter tungsten balls ensured uniform grinding and mechanical disruption of the coal's organic matrix, as evidenced by a 3.5-fold increase in specific surface area from an average of 6.8 to 23.5 m^2/g. After completion of the activation stage, the processed coal was placed in a 3-litre glass reactor equipped with a bubbling system through which an ozone-oxygen mixture at 80 g/m^3 was fed for 1 hour. The ozonation temperature was maintained at 25°C, and the gas flow rate was 1.5 l/min. This treatment resulted in partial oxidation of the carbon particle surface, as evidenced by an increase in the content of oxygen-containing functional groups from 5.3 to 8.1 wt%. Next, thermal dissolution in a tetralin medium was performed. For this, 20 g of activated carbon was mixed with 100 ml of solvent and loaded into a Parr 4560 autoclave with a Teflon liner. The mixture was heated to the required temperature at a rate of 5°C/min. In the first series of experiments, the holding temperature was set at 400°C, the process duration was 90 minutes, and the stirring speed was 200 rpm. In the second series of experiments, the temperature was varied from 375 to 425°C in 25 °C increments. Additionally, a series of experiments was conducted in which ultrasonic treatment was initially performed using a Hielscher UP400S unit. Ultrasonic treatment at 24 kHz and 300 W was performed for 30 minutes immediately before loading the sample into the autoclave. After thermal dissolution was complete, the autoclave was cooled to 50°C, the pressure was reduced to atmospheric, and the reaction mixture was filtered to separate the liquid and solid phases. Liquid products were subjected to vacuum distillation at a residual pressure of 10 kPa to isolate low-boiling (up to 220°C) and medium-boiling (220–350°C) fractions. The remaining heavy residue was analysed for resin and hydrocarbon compounds. The experimental results showed that the degree of conversion of coal organic matter at 400°C and standard activation was 78.2%. At 425°C, the conversion increased to 84.6%, but this was accompanied by an increased content of high-boiling resins and partial thermogenic destruction of tetralin, as evidenced by the appearance of benzene compounds in the light fraction. Ultrasonic treatment, compared with the control series, showed a significant effect. On average, the specific yield of liquid products in the temperature range of 375–425°C fluctuated from 48.1 to 57.9% of the dry coal weight. The heating rate also had a significant effect: with an increase in the heating rate from 3 to 10°C/min, the yield of liquid fractions decreased by an average of 7.2%, which was associated with the formation of a denser shell on the surface of the coal particles and the penetration of the solvent into their pores. Analysis of the group composition of the dissolution products showed that the low-boiling fraction contained, on average, 54% alkyl aromatic compounds, about 20% saturated hydrocarbons, and 26% oxygen-containing components. The medium-boiling fraction consisted mainly of polycyclic aromatic hydrocarbons (65–70%) and resinous compounds (about 25%). The residual carbon content in the heavy residues ranged from 38 to 45 wt.%. Additional elemental analysis of the liquid fractions showed 85.6% carbon, 8.9% hydrogen, and 5.5% oxygen. These data indicate that the hydrocarbon components obtained are of sufficiently high quality for subsequent use as raw materials in binder or fuel production. For comparison, additional thermal dissolution tests were carried out without prior mechanochemical activation. Under these conditions, the conversion of organic matter was only 61.4% at 400°C and 64.9% at 425°C, which are the values obtained under combined treatment. This confirms the importance of the mechanical and ozone-activation stages—a comparative analysis that is even superior to those reported. For example, in studies by Luik et al., liquid product yield at comparable temperatures did not exceed 50–55%, and conversion ranged from 60 to 70%. In the works of Li et al., at a dissolution temperature of 400°C in tetralin, only 72% conversion was obtained, which is 6% lower than that achieved with a combination of mechanochemical activation and ultrasonic treatment. The results also showed that ultrasonic activation increased the yield of light hydrocarbons and products across fractions. During ultrasonic treatment, the proportion of polycyclic aromatic hydrocarbons in the medium-boiling fraction decreased from 70 to 61%, indicating a milder mode of coal matrix destruction. The proportion of polycyclic aromatic hydrocarbons in the medium-boiling fraction decreased from 70% to 61%, indicating a milder mode of coal-matrix destruction. The use of various temperature modes of 400-410 °C, at which the maximum conversion is achieved without substantial thermogenic destruction. An optimal dissolution temperature range of 400-410 °C was identified, at which the maximum conversion is accomplished without significant thermogenic degradation of the solvent. When analysing the heating rate, it was found that heating at a minimum rate of 3 °C/min increased the yield of liquid products to 58.6%, due to a longer solvent effect on the coal mass within the active temperature zone. By contrast, at a heating rate of 10 °C/min, conversion did not exceed 80.3%, and the proportion of heavy residues increased (Table 1). These data products. In this context, recent thermal dissolution/direct coal liquefaction studies at ≈400–425 °C typically report conversions of around 60–75% and liquid product yields of ≤55% under single-step pretreatments or without pretreatment. By contrast, our integrated activation achieved 88.3% conversion and up to 57.9% liquid yield under otherwise comparable temperature windows [22, 23, 24]. These data place the present workflow at the upper end of reported performance while maintaining donor-solvent integrity through moderated heating rates. A comparative analysis of the physicochemical properties of liquid products obtained under different conditions showed that the density of the low-boiling fraction ranged from 0.89 to 0.93 g/cm³, the average heat of combustion was 40.5 MJ/kg, and the viscosity at 20 °C ranged from 2.5 to 3.2 mm²/s. These indicators indicate the potential for using the products as components of fuel mixtures (Table 2). Additional experiments varying the holding time at 400 °C from 60 to 120 minutes showed that a 120-minute holding time increased the degree of conversion to 90.4%. However, the proportion of high-boiling resins also rose to 32%. Therefore, the optimal holding time was about 90 minutes. To assess product stability, some liquid fractions were stored at 25 °C for 25 days. After a month, an increase in the acid number from 2.4 to 3.1 mg KOH/g and a slight rise in viscosity by 0.3 mm²/s were observed, which may be associated with polymerisation of the resinous components. In general, the results demonstrate the high efficiency of mechanochemical and ozone activation, combined with thermal dissolution, in increasing the yield of valuable liquid products. The systematic variation of temperature, holding time, heating rate, and preliminary treatment enabled the determination of the optimal process conditions that yield the maximum conversion of coal organic matter to liquid fractions with specified properties. Determination of the (Table 3). Comparison of the data with results from similar studies by foreign and domestic authors showed that combining mechanochemical action with low-temperature ozonolysis enables the production of higher-quality liquid hydrocarbons in greater volumes than traditional thermal dissolution methods (Table 4). These data can serve as a basis for further scaling of the process and the development of industrial technologies for the complex processing of coal to produce synthetic fuels and chemical products.

Table 1 The Effect of Temperature and Holding Time on the Degree of Conversion and Yield of Fractions.

Conversion rate, %	Density, g/cm^3	Degree of conversion, %	Yield of the low-boiling fraction, % by weight	Yield of the medium-boiling fraction, % by weight	Residue, % by weight
375	90	72.3	44.5	19.1	36.4
400	90	78.2	48.7	22.5	29.3
400	120	90.4	47.9	32.0	19.6
425	90	84.6	46.2	28.4	25.4

Table 2 The Comparison of the Properties of Liquid Products Obtained under Different Activation Modes.

Processing mode	Conversion rate, %	Density, g/cm^3	Heat of combustion, MJ/kg	Content of aromatic hydrocarbons, %	Viscosity at 20 °C, mm^2/s
Without activation	64.9	0.91	39.7	58	3.4
Mechanochemical activation	78.2	0.90	40.1	62	3.1
Mechano- and ozone activation	84.6	0.89	40.5	65	2.8
Mechano-, ozone and ultrasound	88.3	0.89	41.2	68	2.7

Table 3 The Influence of the Heating Rate on the Distribution of Fractions and Quality Indicators of Products.

Heating rate, °C/min	Conversion rate, %	Low-boiling fraction, % mass	Medium-boiling fraction, % mass	Residue, % mass	Heat of combustion, MJ/kg	Viscosity, mm^2/s
3	89.1	58.6	24.7	16.7	41.2	2.6
5	78.2	48.7	22.5	29.3	40.5	2.8
10	80.3	45.3	21.4	33.3	39.8	3.2

Table 4 The Comparison of the Results of this Work with Data from Similar Studies.

Source	Coal (deposit)	Temperature, °C	Degree of conversion, %	Yield of liquid products, % by weight.	Activation methods used
The present study	Barzas	400	88.3	57.9	Mechanochemical, ozone, ultrasound
Luik et al. [18]	Tallinn shale	400	69.5	51.3	Without activation
Li et al. [19]	Shenfu	400	72.0	54.2	Mechanical
Zhang et al. [20]	Huolingol brown coal	400	70.4	52.1	Thermal oxygen treatment
Tiikma et al. [21]	Australian stone	400	75.2	55.0	Mechanochemical + low-temperature oxygenation

4.CONCLUSION

The study confirmed the high efficiency of the combined approach to thermal dissolution of coal from the Barzas deposit, comprising mechanochemical activation, ozone oxidation and ultrasonic treatment. The results showed that this combination maximised the conversion of coal organic matter and enabled the targeted regulation of the composition of liquid products. At 400 °C, with preliminary ultrasonic activation, conversion reached 88.3%, significantly exceeding the dissolution indicators without activation (64.9%) or with only mechanochemical activation (78.2%). At the same time, the yield of liquid products increased to 57.9% of the dry coal mass, a notable improvement compared with similar studies, in which this figure typically did not exceed 55%. Of particular importance was the study of the effect of process temperature. It was found that at 400–410 °C, an optimal balance between high conversion and minimal solvent thermogenic destruction was achieved. As the temperature reached 425 °C, conversion increased to 84.6%; however, the contents of polycyclic aromatic hydrocarbons and resinous components also increased, thereby degrading the quality of the liquid fractions. A crucial factor in the process was the heating rate. At a minimum accelerated at an accelerated heating rate of 3 °C/min, the conversion was 89.1%, and the yield of light fractions was 58.6%. Accelerated heating to 10 °C/min decreased conversion to 80.3% and increased the proportion of residual carbon. These data demonstrate that strict control of the thermal regime is a key condition for effective dissolution. Analysis of the physicochemical properties of the liquid products showed that the density of the low-boiling fraction component. The heat of combustion was 41.2 MJ/kg with combined activation, indicating the potential for using these products as components of fuel mixtures. The content of aromatic hydrocarbons in the light fraction increased from 58% in the control experiments without activation to 68% after complex treatment, indicating deeper destruction of the carbon matrix and increased extraction of valuable organic compounds. At the same time, the acid number of the liquid fractions after a month of storage increased only from 2.4 to 3.1 mg KOH/g, and the viscosity increased by 0.3 mm²/s, indicating acceptable stability of the obtained products. A comparative analysis of the results with literature data confirmed that the chosen methodology was superior to traditional dissolution schemes for tetralin, yielding higher yields and better-quality liquid hydrocarbons. For example, conversion was only 72%, whereas in this study, under similar conditions, it was up to 88.3%. Therefore, the combination of mechanochemical destruction of the coal structure, ozone oxidation and ultrasonic activation not only increased the degree of conversion of organic matter but also enabled purposeful control of the composition and properties of liquid fractions, creating the prerequisites for their further use in the production of synthetic fuels and organic binders. Translation to pilot and industrial scales is feasible using continuous equipment that preserves the residence-time and heating-rate controls identified here. For pretreatment, ozone contactors should ensure gas–solid destruction and the safe destruction of off-gas. Ultrasonic energy delivery can be implemented using in-line high-power probes or hydrodynamic cavitation. For dissolution, moving from a stirred batch autoclave to a continuous tubular or loop reactor will require attention to heat-transfer coefficients, solids handling, and fouling control. A closed solvent loop with distillation, hydrogen-donor balance management (tetralin ↔ naphthalene cycling), and make-up hydrogenation is essential. Foreseeable challenges include ozone safety and materials compatibility, scalable solids feeding, and minimising solvent cracking at the upper end of the temperature window. Nonetheless, the integrated activation route provides clear operating targets (400–410 °C, moderate heating rates, ~90 min dwell time) for pilot-plant design.

CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

A.S. Kasimov: Conceptualization, Methodology, Investigation, Formal analysis, Data curation, Writing – original draft. G.N. Ibragimova: Validation, Resources, Visualization, Writing – review & editing, Project administration. I.V. Gordienko: Supervision, Methodology, Writing – review & editing, Funding acquisition. A.V. Masenko: Experimental setup, Data acquisition, Investigation, Visualization. A.A. Aleksandrov: Software, Data processing, Statistical analysis, Writing – review & editing.

DECLARATION OF COMPETING INTEREST

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

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