JOURNAL OF SUSTAINABLE MATERIALS PROCESSING
AND MANAGEMENT
e-ISSN: 2821-2843

Vol. 5 No. 2 (2025) 74-82
https://publisher.uthm.edu.my/ojs/index.php/jsmpm

JSMPM

Influence of Temperature on the Rheological Properties of Composite Suspensions Based on Pyrocarbon

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DOI: https://doi.org/10.30880/jsmpm.2025.05.02.006

Article Info

Received: 16 June 2025
Accepted: 29 September 2025
Available online: 30 October 2025

Keywords

Apparent viscosity, composite fuel, pyrocarbon, temperature dependence, shear rate, rheological properties

Abstract

The disposal of used automobile tires is a significant environmental challenge. In industrialized countries, the average annual number of discarded tires per person is approximately one. One of the most common methods of tire recycling is pyrolysis. The solid residue of this process is pyrocarbon. Due to its high dispersity, the presence of numerous submicron carbon particles, and significantly greater porosity compared to coal, pyrocarbon is a promising additive for the production of composite slurry fuel. Such fuel systems are influenced by various external factors, particularly temperature, which alters their properties. It has been established that increasing the temperature from 10°C to 20°C leads to a sharp decrease in the effective viscosity of the medium. Further temperature increases result in a less pronounced reduction in viscosity. In the range of 40–50°C, a slight acceleration in the rate of change of rheological characteristics is observed in the studied composite suspensions. At shear stress levels up to 20 Pa, the viscosity of the suspension decreases sharply due to the intensive breakdown of structural bonds. At 30–60 Pa, the system behaves as a Newtonian fluid with constant viscosity, indicating complete destruction of the structural network. It has been shown that as the temperature rises from 20°C to 40°C, the degree of thixotropy increases, reflecting the system's strong ability to recover through enhanced particle interactions. However, at 50°C, thermal degradation of spatial structures occurs. The established performance characteristics of the obtained suspensions make them suitable for use as liquid fuel in fuel-fired boilers.

1. Introduction

The disposal of used automobile tires remains a pressing environmental issue. In industrialized countries, the average annual number of discarded tires per capita is approximately one. One of the most widespread methods of tire recycling is pyrolysis [1, 2], during which rubber components are converted into mixtures of volatile and liquid thermodestruction products. Essentially, tires are made from vulcanized natural rubber and styrene-butadiene rubber (accounting for 41–45%), steel (13–15%), carbon black (around 28%) and polymer fibers, organic and/or inorganic compounds (14–17%), namely, accelerators and antioxidants like sulfur, zinc oxide and silica [3].

The solid residue of pyrolysis, known as pyrocarbon, accounts for about 30% of the initial feedstock mass. Pyrocarbon primarily consists of amorphous carbon, has a porous structure, and is commonly used as a filler in rubber production.

However, its properties are unstable and depend on both the composition of the feedstock and the pyrolysis conditions. Achieving a product with consistent characteristics during thermodestruction is challenging [4]. Moreover, pyrocarbon often contains a significant amount of inorganic ballast impurities, which limits its practical application. At the same time, composite slurry fuel (CSF) can serve as an independent energy source or be incorporated into coal-water slurries. This type of fuel presents a viable alternative not only to coal but also to liquid (e.g., fuel oil, diesel) and gaseous fuels [5]. An additional advantage is the possibility of using liquid distillates—water-organic mixtures extracted from the pyrolysis process—as a dispersion medium. Preparing slurry fuel based on these distillates prevents their discharge into industrial and domestic water systems and enhances the energy value of the fuel suspensions [6]. Due to its high dispersity, the presence of submicron carbon particles, and significantly greater porosity compared to coal, pyrocarbon is a promising additive for CSF production. Its advantages include a high carbon burnout rate (residue < 0.04%), reduction of atmospheric pollutants (SOx, NOx). This is the enjoyment of the catalytic activity of finely dispersed carbon [6-8]. To be effectively used in energy-generating systems, CSF must meet several technological and operational requirements, including: effective viscosity not exceeding 1.5–1.6 Pa·s; sedimentation stability of 2–3 days for immediate combustion and 10–14 days for transportation and storage.

Temperature is one of the key external factors influencing the rheological properties of composite suspensions. Its impact must be considered in the context of CSF preparation, transportation, storage, and utilization. Typically, fuel preparation occurs indoors, where sub-zero temperatures are not observed during winter [9]. It is known that water viscosity decreases by a factor of 1.8 when the temperature rises from 0 to 20°C [10].

The rheological behavior of coal-water slurries based on coal of varying metamorphic grades has been studied extensively [11–13].

For instance, research on anthracite-based slurries shows a fivefold decrease in viscosity as temperature increases from 3 to 20°C, and a further 30–35% reduction between 20 and 50°C [11]. In work [12], it is shown that when heated from 293 K to 333 K (20-50°C), the dynamic viscosity of the composite suspension decreases by 17-20%. This effect gives an increase in the torch flame by 21-29%. The formulation of composite fuel using Comma Xtech 5W-30 oil (49 wt.%), water (10 wt.%), and long-flame gas coal (40 wt.%) ensures stable viscosity-temperature parameters under various loads and temperatures ranging from 20 to 70°C [13].

Thus, the temperature dependence of rheological properties in coal-water slurries is well documented. Within the framework of the development of recycling technologies, the search for alternative liquid fuels is relevant. The use of pyrocarbon as a carbon-containing material for the production of composite suspension fuels will help solve a number of environmental problems.

The development of CSF based on carbon-containing materials, particularly pyrocarbon, remains insufficiently explored. The aim of this study is to investigate the rheological properties of composite slurry fuel based on pyrocarbon and to examine the influence of temperature and solid phase concentration on its effective viscosity and structural behavior.

2. Materials and Methods

The study involved analyzing the chemical composition of samples, identifying surface functional groups, and determining the technical and structural–sorption characteristics of pyrocarbon, the solid fraction obtained from the pyrolysis of automobile tires.

Pyrocarbon was obtained by pyrolysis of passenger car tires. The technical parameters of pyrocarbon derived from tire pyrolysis were assessed according to DSTU EN standards and are presented in Table 1. The oxide-based component content was determined following DSTU 9045:2020. Elemental composition analysis of the pyrocarbon sample was performed using a Leco CS 230 analyzer in accordance with DSTU EN 15289:2013 and DSTU EN 15104:2013, with results shown in Table 2.

The chemical composition of pyrocarbon surface groups was examined using infrared spectroscopy (IR) on an Avatar 370FT-IR spectrometer (Thermo Nicolet) within the range of 4000–400 cm^{-1}. Samples were prepared as extracts in iso-C_3H_7OH, ortho-$C_6H_4(CH_3)_2$, and C_7H_{14}, with a carbon concentration of 10% relative to the total system. Mass loss during extraction was 11.97%, 14.0%, and 10.87%, respectively. Component identification was performed by comparing the obtained spectra with reference and standard spectra from the instrument's database (EZ Omnic v. 7.0) and literature sources [14].

Microphotographs of pyrocarbon and anthracite were taken using the Levenhuk Rainbow 2L PLUS Lasur optical microscope at 100x magnification.

Structural–sorption characteristics of pyrocarbon mesopores (2–50 nm), including specific surface area *(S_sp)*, pore volume *(V_pore)*, and effective pore diameter *(d_pore)*, were determined using the DFT method based on low-temperature nitrogen adsorption (77 K) on a Quantachrome Autosorb analyzer, with data processed using Quantachrome Instruments software v. 3.2.

The quantity of surface functional groups — $COOH^-$, OH^-, and >C=O — was determined via Boehm titration using 0.05 M NaOH, Na_2CO_3, and $NaHCO_3$ solutions [15]. The total amount of basic groups was determined according to and amounted to 0.053 mg-eq/g [16].

In the second stage, the influence of temperature on the rheological properties of the suspensions was investigated. Composite slurry fuel (CSF) was prepared by homogenizing pyrocarbon particles (200 μm fraction) with a dispersion medium consisting of technical water and chemical reagents. Mixing was performed for 30–40 minutes using a paddle mixer, followed by homogenization in a cavitation disperser. During cavitation treatment, hydrodynamic cavitation occurs in the gap between the rotor and stator, characterized by the formation of microbubbles (up to 25 μm) in the liquid flow and their subsequent collapse via radial compression [17]. This process concentrates high-density cumulative energy at a focal point, enabling the production of oxygen-saturated composite slurry fuel with particle sizes down to 30 μm, stable for up to one month. Several CSF samples were obtained with solid phase concentrations ranging from 51.7% to 60%.

The chemical reagents used included: C-3: a condensation product of β-naphthalene sulfonic acid and formaldehyde (TU 5870-005-58042865-05) OP-10: a product of mono- and dialkylphenol ethoxylation with a degree of ethoxylation of 10. Key rheological parameters of the composite slurry fuel — *the apparent* viscosity (η_{ap}) and shear stress (τ) — were measured using the Rheotest-2 instrument in a thermostated environment with coaxial smooth cylinders (S1/S2 measurement system) at 12 fixed rotor speeds ranging from 0.278 to 243 rpm, corresponding to shear rates (D_r) from 1.0 s^{-1} to 437.4 s^{-1}. For comparative analysis of coal-based dispersion systems, a shear rate of $Dr = 9\ s^{-1}$ is commonly used, and the corresponding viscosity is referred to as the *apparent* viscosity, reflecting the load during preparation and pipeline or tank transportation.

Thermostating was performed using a U15C liquid circulation thermostat (MLW series, Germany) for 5 minutes. Rheological measurements at each temperature were conducted with a fresh portion of CSF, pre-mixed at the target temperature for 0.5 minutes. Temperature regimes ranged from 10°C to 50°C, with a uniform step of 10°C.

3. Results and Discussion

The study of the rheological properties of composite suspension fuels requires a comprehensive study of the structure, technical characteristics, and surface groups of pyrocarbon.

The structural–sorption characteristics of pyrocarbon mesopores (2–50 nm) were determined using the DFT method based on low-temperature nitrogen adsorption at 77 K. The specific surface area was $S\_sp = 35.3\ m^2/g$, the specific pore volume $V\_pore = 0.0365\ cm^3/g$, and the effective pore diameter $d\_pore = 26.8$ nm. It is shown that the pyrocarbon sample has a developed specific porous surface with mesopores.

The number of surface functional groups: COOH - 0.0085 mg-eq/g; OH - 0.0206 mg-eq/g; >C=O - 0.00955 mg-eq/g) was determined by titration according to Boehm, the number of basic groups has determined and has 0.053 mg-eq/g. The data indicate a heterogeneous surface of pyrocarbon with acidic and basic centers with a predominance of the latter.

The definition of the technical characteristics of pyrocarbon is shown in Table 1. From Table 1 it can be seen that the sample was classified as a medium-ash material, has a high content of volatile substances and a high heat of combustion.

Table 1 *Technical characteristics of pyrocarbon*

Object of study	Component	Component content	Method of determination
Pyrocarbon	Humidity Wa, mass%	2,38	DSTU EN 14774-1
	Ad, %	19,5	DSTU EN15403, ISO 1171
	Volatile substances Vdaf, mass%	18,3	DSTU EN15148:2012
	Higher heat of combustion in the dry state Qc, kJ/kg	27959	DSTU ISO 1928, calorimeter IKA C2000
	Higher heat of combustion for the working state Q_p, kJ/kg	27172	

Among the identified metal compounds, the highest concentrations were observed for oxides of SiO_2, ZnO, and MgO. Zinc is commonly used in tire manufacturing in the form of zinc oxide, serving as an activator in sulfur vulcanization and as a stabilizer to prevent rubber degradation. Calcium and magnesium oxides are employed as fillers, while SiO_2 corresponds to white carbon black. The presence of Fe in pyrocarbon is likely due to residual steel cord from worn tires. Since metal cord removal was performed via magnetic separation, the iron content in the pyrocarbon is minimal. A slightly elevated sulfur content was also observed, which is consistent with its role in vulcanization processes.

Table 2 *Elemental analysis of pyrocarbon*

Content of the component in the ash, wt.%								Elemental analysis, % in daf				
SiO_2	ZnO	MgO	Fe_2O_3	Al_2O_3	PbO	CuO	K_2O	C	H	N	O	S
51.51	16.50	13.36	1.50	3.19	0.10	1.23	0.9	77.39	2.81	0.12-0.17	2.6-3.5	2.4

Pyrocarbon exhibits a heterogeneous structure resembling graphitic carbon, but with a lower degree of structural order. One of the carbon modifications present in pyrocarbon is turbostratic graphite — a form in which graphite layers are randomly oriented relative to one another, distinguishing it from crystalline graphite. As a result, pyrocarbon possesses unique physicochemical properties, including high mechanical strength and chemical resistance.

Its structure contains both turbostratic graphite domains and amorphous carbon regions [18], the latter contributing to the material's porosity. As shown in Fig. 1, pyrocarbon (image a) demonstrates a porous architecture with a well-developed specific surface area (S_sp = 35.3 m^2/g) and pronounced surface roughness compared to anthracite coal of grade "A." The particles exhibit both spherical and irregular morphologies.

In contrast, anthracite (image b) predominantly features a crystalline structure with residual amorphous carbon phases. This imparts high density and brittleness, along with a characteristic luster.

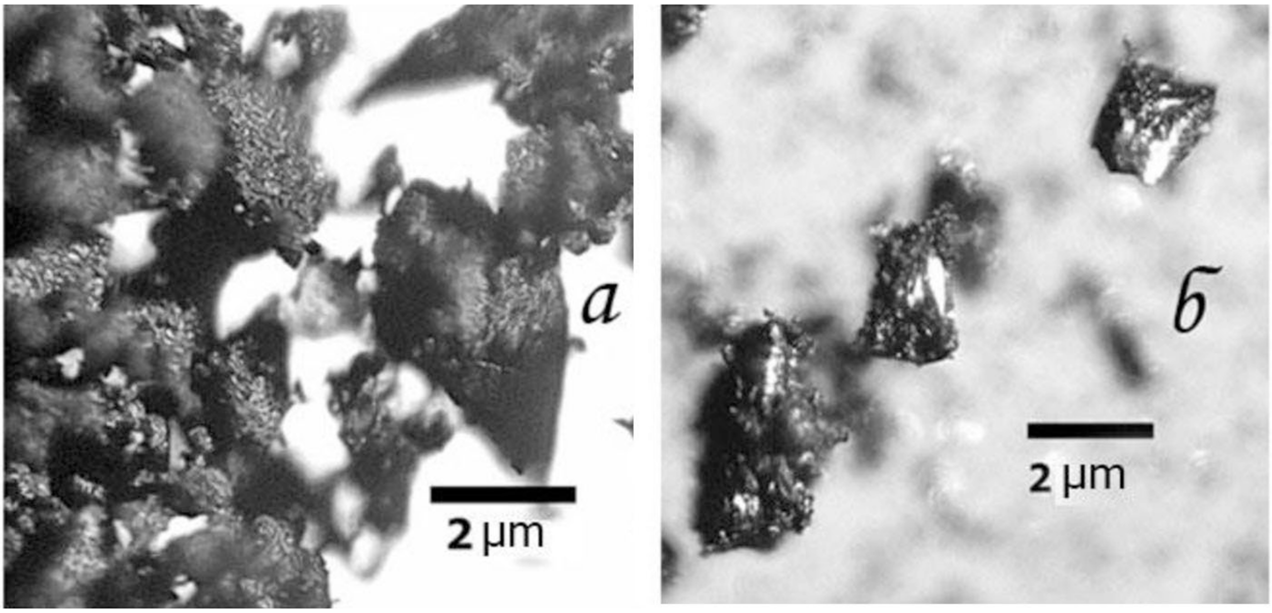


Fig. 1 *Microphotographs of particles: (a) Pyrocarbon; (b) Anthracite*

The presence of amorphous structure and partial graphitization in pyrocarbon was identified using molecular infrared (IR) spectroscopy and examined in detail in previous studies [19]. The IR spectrum of pyrocarbon exhibits absorption bands characteristic of aliphatic compounds (2925 cm^{-1}, 2856 cm^{-1}, 1456 cm^{-1}, 1376 cm^{-1}, 754 cm^{-1}), carbonyl groups (1703 cm^{-1}), aliphatic sulfides (1076 cm^{-1}, 699 cm^{-1}), as well as weakly expressed bands associated with aromatic compounds (1630 cm^{-1}, 1032 cm^{-1}, 877 cm^{-1}).

The viscosity and fluidity of composite liquid fuels, along with the particle size distribution, sedimentation stability, and dispersion of the solid phase, are important factors determining the technology of their transportation, storage, ignition, and combustion. The identified factors are evaluated when analyzing the rheological characteristics of these fuel compositions, which were performed in previous studies [19].

Composite slurry fuels (CSFs) are influenced by various external factors, particularly temperature, which alters their rheological properties. Therefore, this study focused on investigating the temperature dependence of the rheological behavior of CSFs based on pyrocarbon.

To assess the relationship between rheological parameters and temperature, the effective viscosity of CSFs containing pyrocarbon was measured within the range of 20–50°C, for samples with solid phase concentrations from 51.7% to 60 wt.% (Fig. 2). As shown in Fig. 2, the highest effective viscosity was observed at 10°C, which decreased several-fold by 20°C. For CSFs with 60 wt.% solid content, this difference was more pronounced.

Between 20°C and 40°C, a slight decrease in viscosity was recorded across all CSF samples. However, at temperatures of 50°C and above, rheological parameters increased for all compositions. A similar temperature dependence has been reported for CSFs based on lignite [12], whereas anthracite-based CSFs exhibit a continuous decrease in viscosity throughout the 10–70°C range [11]. This behavior may be attributed to the structural characteristics of the carbonaceous carrier in the CSF.

From a technological standpoint, the most optimal temperature range for CSF preparation is 15–35°C, within which pyrocarbon-based CSFs demonstrate a consistent tendency toward reduced effective viscosity.

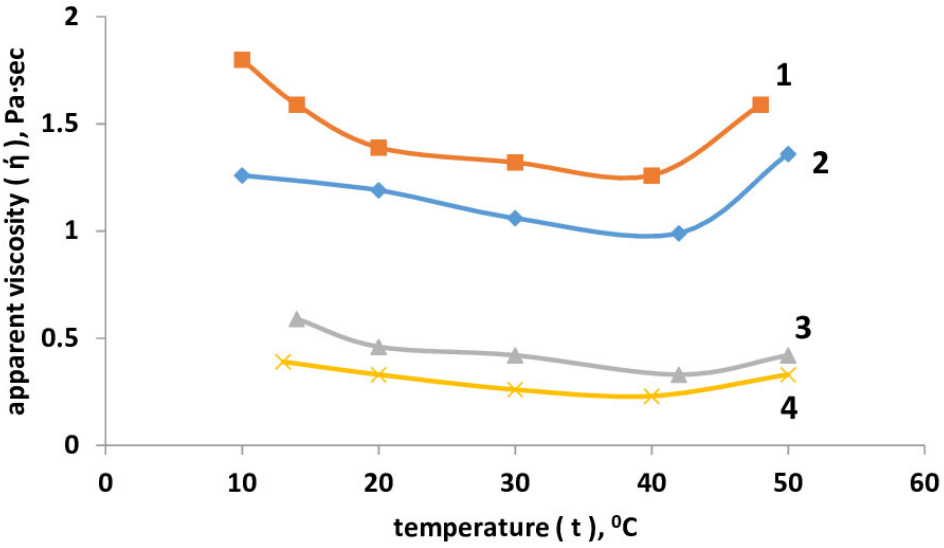


Fig. 2 *Dependence of apparent viscosity (η) on temperature (t) for CSFs with varying solid phase concentrations:1 – 60%, 2 – 58%, 3 – 55%, 4 – 51.7%*

The observed changes in effective viscosity under the influence of temperature may be attributed to several factors:

- The effect of temperature on Brownian motion of anisotropic particles within the suspension;
- Temperature-induced changes in particle aggregation state;
- Modification of the solvation layer surrounding suspended particles.

The increase in viscosity at elevated temperatures is primarily due to the intensification of particle–particle interactions, especially via hydrophobic contact points. For CSFs with solid concentrations of 40–55%, this effect is minimal, owing to the sufficient free volume of the dispersion medium. However, at a solid phase concentration of 60%, temperatures exceeding 40°C led to enhanced particle contacts and reduced water content due to evaporation, resulting in a noticeable rise in viscosity beyond 40°C.

Fig. 3 presents the complete rheological curves for CSFs with a solid phase concentration of 58%. The curves exhibit a concave shape, indicating on pseudoplastic behavior of the system. As temperature increases, the slope of the curves relative to the shear stress axis decreases, reflecting structural densification caused by intensified particle interactions and a reduction in the interstitial dispersion medium within the suspension's framework.

Similar rheological curve profiles were observed for CSFs with 55% and 60% solid phase concentrations. The trend in slope reduction with increasing temperature remains consistent across these compositions.

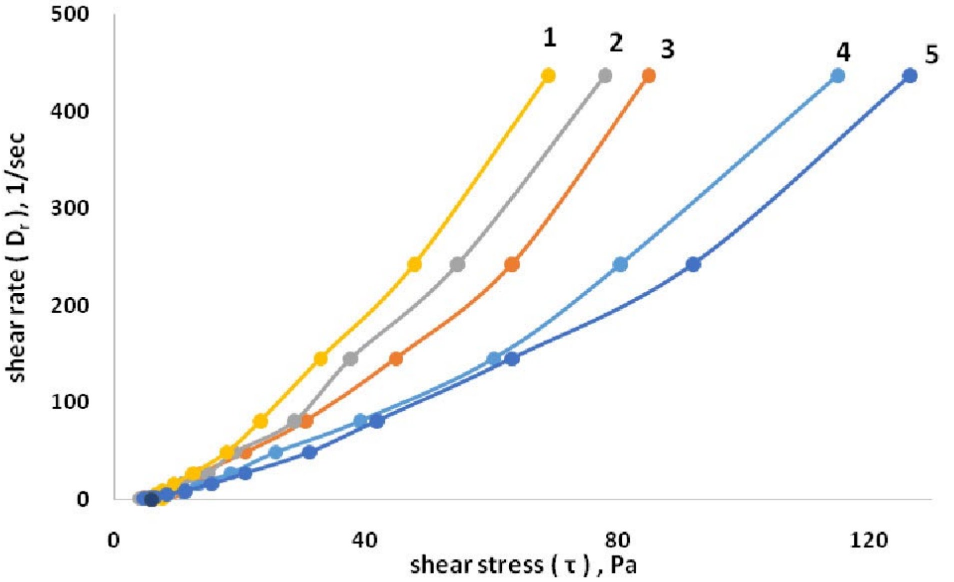


Fig. 3 *Dependence of shear rate (Dr) on shear stress (τ) for CSFs at different temperatures: 1 – 50°C, 2 – 40°C, 3 – 30°C, 4 – 20°C, 5 – 10°C*

Fig. 4 illustrates that the rheological curves exhibit a concave shape at temperatures of 20°C, 40°C, and 60°C, indicating a pseudoplastic flow behavior. As shear stress increases at a given temperature, the viscosity initially drops sharply, followed by a more gradual decline until it reaches a minimum value. Beyond this point, viscosity remains constant.

In the shear stress range up to 20 Pa, the viscosity of the suspension decreases rapidly, reflecting intensive breakdown of structural bonds within the system. At 30–60 Pa, the suspension behaves as a Newtonian fluid with constant viscosity, corresponding to complete destruction of the structural network. Within this region, the structure undergoes near-total breakdown during flow, with only minimal recovery.

This rheological pattern was consistently observed across all composite slurry fuels at the studied temperatures.

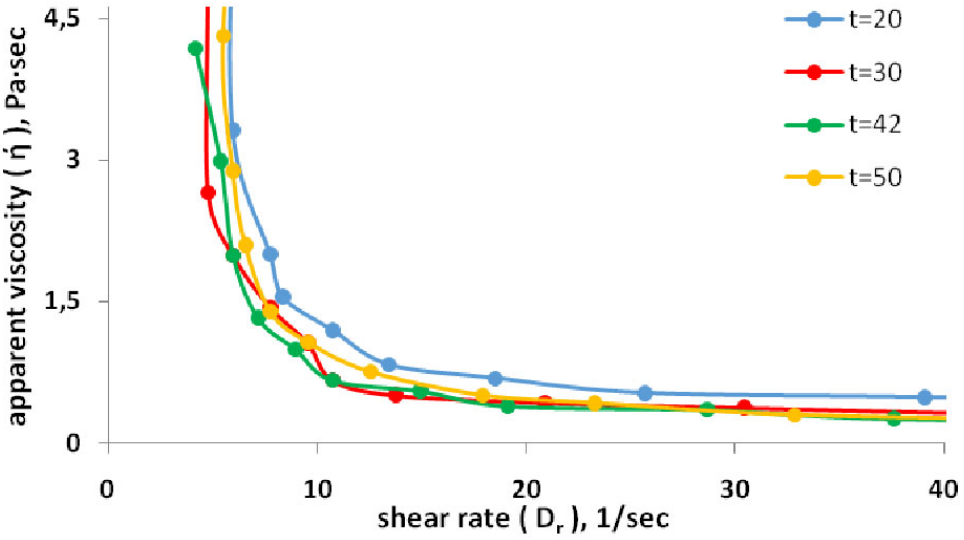
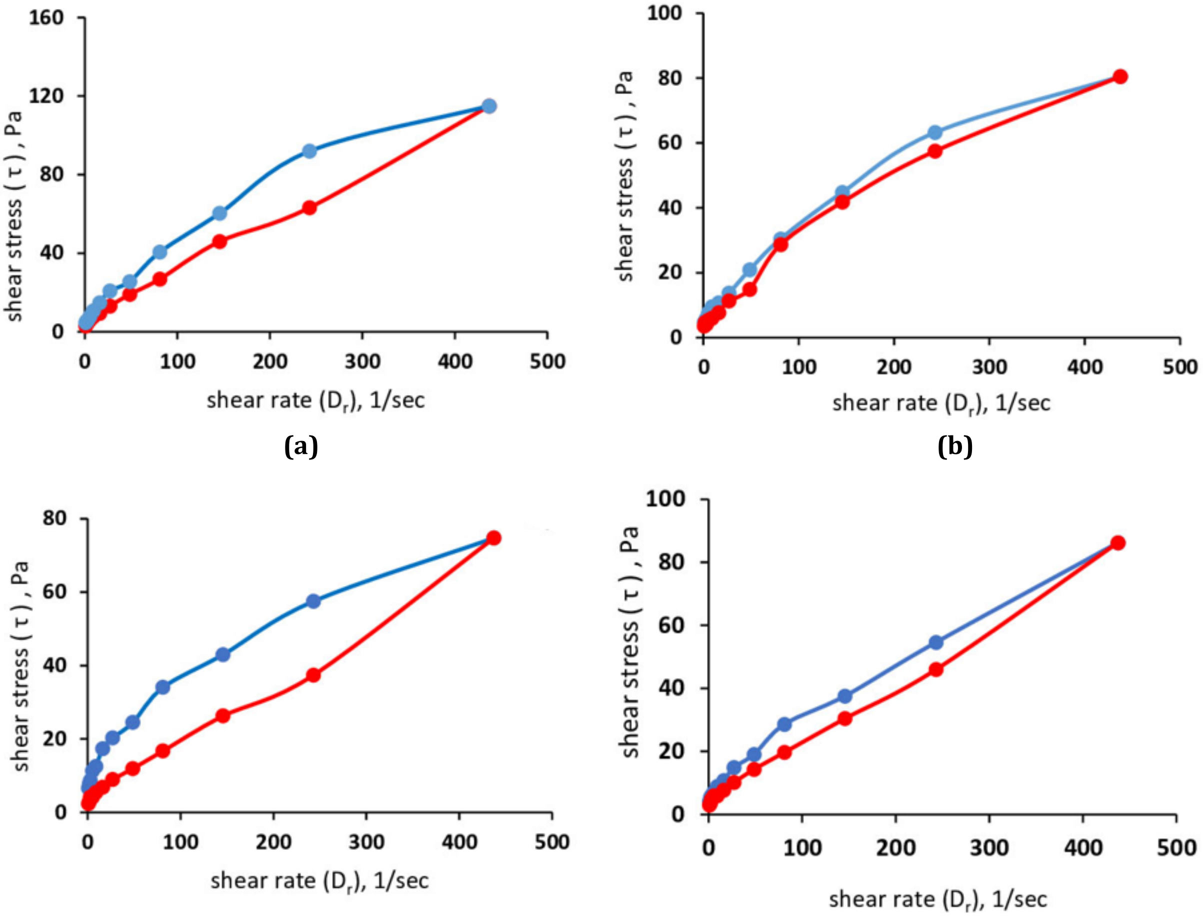


Fig. 4 *Dependence of viscosity (η) on shear stress (τ) at different temperatures: 20 °C, 30 °C, 42 °C, 50 °C*

Experimental results revealed that the structure of composite slurry fuels (CSFs) can recover over time following mechanical disruption. During shear deformation, particle–particle contacts within the dispersed phase are broken. Upon removal of the load, these contacts are gradually restored due to the mobility of the dispersion medium and the Brownian motion of solid-phase particles.

Thixotropic behavior in CSFs was observed as a decrease in viscosity under mechanical stress, followed by a gradual increase once the stress was removed. As shown in Fig. 5, the shear stress–shear rate relationship for pyrocarbon-based suspensions demonstrates recovery of the coagulated structure after unloading. Under applied shear, the system breaks into discrete fragments; when the load is removed, the structure re-forms, producing a hysteresis loop indicative of system structuring and thixotropy. The area of the hysteresis loop serves as a quantitative measure of the degree of thixotropy.



(a)

(b)

(c)

(d)

Fig. 5 *Dependence of shear rate (Dr) on shear stress (τ) for CSFs at different temperatures: (a) 20°C, (b) 30°C, (c) 40°C, (d) 50°C (Blue curve – increasing Dr; red curve – decreasing Dr)*

It was found that for CSFs exhibiting pronounced thixotropic behavior, the flow curve obtained during gradual shear stress increase does not coincide with the curve obtained during gradual stress reduction. For CSFs with a solid phase concentration of 58 wt.%, an increase in temperature from 20°C to 40°C led to a decrease in hysteresis loop area, indicating enhanced structural recovery and stronger particle–particle interactions.

Notably, the flow curves at 50°C exhibited a larger hysteresis loop area compared to those at lower temperatures. This suggests that thermal degradation of spatial structures occurs at elevated temperatures. At 10–20°C, the spatial structures remain highly stable and viscous, resisting mechanical breakdown. A similar trend was observed for CSFs with solid phase concentrations of 55% and 60%.

4. Conclusions

The disposal of used automobile tires is a significant environmental challenge. In industrialized countries, the average annual number of discarded tires per person is approximately one. One of the most common methods of tire recycling is pyrolysis. The solid residue of this process, pyrocarbon, due to its high dispersity, abundance of submicron carbon particles, and significantly greater porosity compared to coal, is a promising additive for the production of composite slurry fuel (CSF).

Its advantages include a substantially higher carbon burnout rate residue temperature, which alters its rheological behavior.

Experimental results demonstrated a sharp decrease in effective viscosity as temperature increased from 10°C to 20°C. Further temperature increases led to a more gradual reduction in viscosity. In the range of 40–50°C, a slight increase in the rate of rheological change was observed across all tested CSFs.

Based on the research conducted for composite suspensions based on pyrocarbon, the optimal temperature range is 15-40°C.

The rheological curves exhibited a concave shape at temperatures between 10°C and 50°C, indicating pseudoplastic flow behavior. At shear stresses up to 20 Pa, viscosity dropped sharply due to intensive structural breakdown. Between 30–60 Pa, the system behaved as a Newtonian fluid with constant viscosity, corresponding to complete destruction of the structural network.

As the solid phase concentration increased from 55% to 60%, the influence of temperature on rheological properties became more pronounced. It was shown that increasing temperature from 20°C to 40°C led to a rise in thixotropy, indicating strong structural recovery and enhanced particle–particle interactions within the pyrocarbon-based system. At 50°C, however, thermal degradation of spatial structures was observed.

The operational characteristics of the obtained CSFs suggest their suitability as liquid fuel for fuel-fired boilers. Considering the availability and relative low cost of the feedstock (solid pyrolysis residues from tire rubber), this type of fuel has the potential to compete successfully with conventional liquid fuels.

Acknowledgement

The authors are thankful to the company Liquid Carbo, headed by Zinin V.V., for the provided samples of pyrocarbon pyrolysis of tires.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Olena V. Shkutkova, Anatolii S. Makarov;* ***data collection:*** *Olena V. Shkutkova; Viacheslav V. Zinin;* ***analysis and interpretation of results:*** *Olena V. Shkutkova; Roman Evh. Klishchenko; Ivan V. Kornienko;* ***draft manuscript preparation:*** *Olena V. Shkutkova, Viacheslav V. Zinin. All authors reviewed the results and approved the final version of the manuscript.*

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