Thermogravimetric kinetic study of solid recovered fuels pyrolysis

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Abstract

In the Republic of Serbia there are significant quantities of coffee and tire wastes that can be utilized as Solid Recovered Fuel (SRF) and used as an additional fuel for co–combustion with coal and biomass in energy production and cement industry sectors. Differences between SRF and base fuel are a cause of numerous problems in design of burners. The objective of this study was to determine the kinetic parameters for the thermochemical conversion of selected SRF using Simultaneous Thermal Analysis (STA). Samples of coffee and tire waste were used for the experimental tests. Thermal analysis was carried out in nitrogen atmosphere at three different heating rates 10, 15 and 20 K/min for each sample, while it was heated from room temperature up to 900 °C. Two sample sizes x <0.25 mm and 0.25 < x <0.5 mm of each SRF were used in experiments, in order to obtain reliable Thermal Gravimetric Analysis (TGA) data for estimation of kinetic parameters for SRF pyrolysis. Experimental results were used for determination of pre-exponential factor and activation energy according to methods presented in the literature. Presented research provides valuable data of coffee and tire waste that can be used for the burners design.

Keywords: *simultaneous thermal analysis; STA; thermal gravimetric analysis; TGA; kinetic parameters; solid recovered fuels; pyrolysis*.

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1. INTRODUCTION

Solid recovered fuels (SRF) and Refuse derived fuels (RDF) are fuels produced from non-hazardous municipal solid waste (MSW) and commercial and industrial (C&I) wastes. European environmental and energy policies include measures to enhance recovery of residual waste as well as to promote energy derivation from biomass and waste [1-3]. Solid biofuels have emerged as a suitable, low-carbon alternative to coal. These can be used in a form of loose chips, pellet or briquette, and can come from a wide variety of sources. The production of waste derived fuels for energy recovery has been a popular waste management option since the 1990's. Many waste fractions which cannot be easily reused or recycled, particularly if they are composed of materials that are difficult to properly sort or separate, may have a high caloric value that can be used as a fuel for energy recovery. SRF is a high quality alternative to fossil fuel and is produced from commercial waste including paper, card, wood, textiles and plastic with a calorific value of 17 – 22 MJ/kg.

Sources for raw materials to produce SRF are first of all: residues from the production processes, industrial wastes, wastes from the selective collection from trade and households, solid municipal waste and construction-overhaul waste [4,5]. For the production of the SRF, heterogeneous industrial waste, as well as mixed wastes can be used (the more components in wastes the more complex is the production process). One can distinguish two major types of SRF: granulated (characterized by variable size of granules) and formed (or molded) (as pellets, cubes and briquettes). SRF produced in this manner and in compliance with a series of European Standards, ensures a consistently high energy content, low moisture and a high proportion of carbon neutral biomass.

There are many researches on the pyrolysis of different SRF all over the world in the last decade due to the raising public awareness of environmental concerns [6,7]. Determination of the kinetic parameters for the thermochemical conversion of selected SRF using simultaneous thermal analysis (STA) is presented in this research. Thermal gravimetric analysis (TGA) is a thermal analysis technique which measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. TGA measurements are used primarily to determine the composition of materials and to predict their thermal stability at elevated temperatures. Samples of coffee and tire waste were used for this experimental research. During the preparation of a coffee

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beverage, raw coffee powder is contacted with hot water or steam under conditions favoring the release of aroma compounds and other coffee-bean constituents into the liquid. From these operations, a solid residue known as spent coffee grounds (SCG) is produced. SCG do not have any commercial value and are usually discarded as solid waste or, to a very limited extent, sent to compost facilities. In recent years, however, the increasing awareness of the need for waste reduction and environmental protection has stimulated the search for possible methods of using this waste [8]**.** Amount of tire waste in the world per year is also enormous. The impact of tire waste on the environment can be minimized by recycling the material or by energy recovery [9,10]**.** Tires are usually made of different types of rubber, including natural rubber (NR), butadiene rubber (BR) and styrene butadiene rubber (SBR), along with other synthetic polymers, carbon black, and some organic additives. Pyrolysis (thermal degradation in the absence of oxygen) is one of the main techniques used in tire energy recovery [11]**.** Pyrolysis of scrap or waste tires (WT) is an attractive alternative to disposal in landfills, allowing the high energy content of the tire to be recovered as fuel. Using the same mass of WT as fuel produces equal energy as oil and 25 % more energy than coal. Some results in literature [12] show that the average kinetic parameters of tire derived fuels are: the activation energy of 1322±244 kJ/mol, and the pre-exponential constant of $2.06\pm3.47\cdot10^{10}$ min^{-1}.

The aim of the research presented in this paper was to obtain reliable TGA data for estimation of kinetic parameters for SRF pyrolysis. Thermal analyses (TA) were carried out in nitrogen atmosphere at three different heating rates for each coffee and tire waste sample.

2. MATERIALS AND METHODS

2.1. Materials and sampling procedure

A sample of coffee waste (SCG) was taken from a local cafe that produces large amounts of this waste material daily. The sample is the residue from the preparation of espresso coffee and is directly collected from the vessel of the appliance. A waste tire sample (WT) is provided as granules produced in a waste tire granulation plant.

After sample collection according to SRPS EN 15442:2012, the SCG and WT samples were prepared in the laboratory according to SRPS EN 15443:2012. These standard procedures were performed in order to form analytical samples, which were additionally sieved in order to obtain two particle size dimensions for each of the samples as $x < 0.25$ mm and $0.25 < x < 0.5$ mm. For each of the samples prepared in this way, all tests related to this research were performed according to the acting standard in order to obtain proximate analysis data. The remaining sample was used for STA testing and the results of these tests were used for further analysis of the kinetic parameters.

2.2. Simultaneous thermal analysis (STA) measurements

For conducting the experiments, STA 449 Jupiter F5 produced by NETZSCH, Germany was used. It is capable of applying two different analyses on the same sample at the same time, TGA and differential scanning calorimetry (DSC). In this way, acquired results are more accurate and more data are obtained in a shorter period of time than conducting those analyses separately.

Using STA, pyrolysis process of two different samples of SRF (SCG and WT) was analyzed, using two different particle sizes for each sample: $x < 0.25$ mm and $0.25 < x < 0.5$ mm.

Alumina crucibles were used for all tests, and each measurement was carried out in nitrogen atmosphere. Each sample was tested using three different heating rates, one for each measurement. It was considered that heating rates of 10, 15 and 20 K/min would be the best for kinetic parameters comparison. Using these heating rates, samples were heated from room temperature up to 900°C and afterwards maintained at the reached temperature for 10 minutes. The final set temperature 900°C was closely but never exactly reached for each measurement (the lower was the heating rate, the closer was the final temperature to 900°C). Precise heating was provided by the Sample Temperature Controller (STC) which ascertained that the referring temperature during the process was the sample temperature, not the furnace one.

Nitrogen was used to provide the inert atmosphere for maintaining the pyrolysis but also as the protective gas for protection of the balance inside the instrument, and the flow rates of both carrier and protective gas were set to 50 ml/min. Internal balance is highly sensitive (0.1 µg) and it is used for measuring the sample (19±4 mg of SCR or WT was used in measurements) and monitoring the sample mass change during the process.

Obtained data from these measurements were further used to estimate kinetic parameters for SRF using selected model-free methods [13].

3. KINETIC THEORY

The Arrhenius equation is generally used to define dependence of the reaction rate constant on temperature:

$$k = Ae^{-\frac{E}{RT}} \tag{1}$$

where *A* is the pre-exponential factor (frequency factor), *E* – the activation energy, *R* – the universal gas constant and *T* – the absolute temperature.

The rate of thermochemical conversion in the kinetic theory is usually described as:

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = k(T)f(\alpha) \tag{2}$$

where α represents the degree of conversion, *t* – time, *k*(*T*) – the reaction rate constant, *f*(*a*)– the reaction model. The conversion α is defined as:

$$\alpha = \frac{m_0 - m_\tau}{m_0 - m_f} \tag{3}$$

Where m_0 is the mass of initial sample, m_τ – the sample mass at time τ and m_f – the mass after pyrolysis. In order to calculate kinetic parameters based on results acquired from TGA, by combining equations (1) and (2), a fundamental analytical equation is obtained:

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = Af(\alpha)e^{-\frac{E}{RT}} \tag{4}$$

Assuming that the heating rate is defined as β = d*T*/d*t*, the eq. (4) can be written as:

$$\frac{\mathrm{d}\alpha}{\mathrm{d}T} = \frac{A}{\beta}f(\alpha)\mathrm{e}^{-\frac{E}{RT}} \tag{5}$$

Estimation of kinetic parameters, such as the pre-exponential factor (*A*) and the activation energy (*E*), which are based on the kinetic theory and TGA data can be made by different techniques. These techniques are known as model-fitting (which could include identification of the kinetic reaction model) or model-free (i.e. isoconversional). In the model-fitting approach, kinetic parameters are determined by assuming the form of *f*(α). Since in a non-isothermal experiment both *T* and α vary simultaneously, the model-fitting approach generally fails to achieve a clear distinction between the temperature dependence, *k*(*T*) and the reaction model *f*(α). As a result, almost any *f*(α) can satisfactorily fit the data at the cost of drastic variations in kinetic parameters, which compensate for the difference between the assumed form of *f*(α) and the true but unknown reaction model. For this reason, the model-fitting methods tend to produce highly uncertain values of kinetic parameters. These drawbacks can be avoided by the use of isoconversional methods. Model-free methods allow the activation energy to be determined as a function of the extent of conversion and/or temperature and this dependence is determined without making any assumptions about the reaction model. This means that these methods are likely to produce consistent kinetic results from isothermal and non-isothermal experiments [14].

3.1. Model – free methods

The Friedman method is the most common differential isoconversional method and it is usually described by the following equation:

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = \beta\frac{\mathrm{d}\alpha}{\mathrm{d}T} = Af(\alpha)\cdot e^{-\frac{E}{RT}} \tag{6}$$

It has to be noted that the analysis does not provide the knowledge of the reaction model. The Friedman method applies the logarithm of the conversion rate as a function of the reciprocal temperature at different conversion degrees, which is obtained by simple rearrangement of the eq. (6):

$$\ln\left(\frac{\mathrm{d}\alpha}{\mathrm{d}t}\right) = \ln\left[\beta\left(\frac{\mathrm{d}\alpha}{\mathrm{d}T}\right)\right] = \ln\left[Af(\alpha)\right] - \frac{E}{RT} \tag{7}$$

It is assumed that the conversion function $f(\alpha)$ remains constant, which implies that degradation is independent of temperature and depends only on the rate of mass loss. A plot of $\ln(\mathrm{d}\alpha/\mathrm{d}t)$ or $\ln[\beta(\mathrm{d}\alpha/\mathrm{d}t]$ *vs.* $1/T$ yields a straight line, the slope of which corresponds to $-E/R$ [13].

The Flynn-Wall-Ozawa (FWO) method is also one of the most commonly accepted methods for computation of kinetic parameters. This method uses the correlation between different heating rates, activation energy and inverse temperature. In this method, the activation energy is determined from the equation:

$$\ln\beta=\ln\left(\frac{AE}{Rg(x)}\right)-5.331-1.052\frac{E}{R}\frac{1}{T} \tag{8}$$

With the assumption that β has a fixed value, E can be determined from the slope of the straight line obtained by plotting $\ln\beta$ *vs.* $1/T$ [13].

The Kissinger-Akahira-Sunose (KAS) method is an integral isoconversional technique. The fundamental expression for calculation of the activation energy by this method is:

$$\ln\left(\frac{\beta}{T^2}\right)=\ln\left(\frac{AR}{Eg(x)}\right)-\frac{E}{RT} \tag{9}$$

By plotting $\ln(\beta/T^2)$ *vs.* $1/T$ for constant α, straight lines are obtained, which slopes are used for evaluation of the activation energy.

Since determination of the frequency factor for model-free methods requires the assumption of the reaction model $f(\alpha)$, in this paper, it is assumed to take the following form:

$$f(\alpha)=(1-\alpha)^2 \tag{10}$$

while the pre-exponential factor was determined for order of reaction $n = 2$.

4. RESULTS AND DISCUSSION

4.1. General and STA results

TGA data obtained from analyzing SRF samples (spent coffee grounds and waste tire) were used for kinetic study using the aforementioned methods (Section 3.1). Proximate analysis results (M – moisture, VM – volatile matter, A – ash, FC – fixed carbon and LHV – lower heating value) of used SRF samples for air dried mass are presented in Table 1.

Table 1. Proximate analysis for analyzed samples

	M^{ad}, % mass	VM^{ad}, % mass	A^{ad}, % mass	FC^{ad}, % mass	LHV^{ad}, MJ/kg
SCG	10.33[a]	70.30	3.02	16.35	17.30
WT	0.64	56.98	16.28	26.10	32.05

[ad] On a air-dried basis

In order to obtain TGA data required for kinetic parameters determination, STA analyzer was used. Those data are presented on following diagrams as SRF sample conversion (α) or mass change rate ($\mathrm{d}\alpha/\mathrm{d}t$) as a function of temperature (T). Results for SCG are presented in Figures 1 and 2 for different sample sizes, while the WT results are presented in Figures 3 and 4.

Experimental results of TGA presented in Figures 1-4 show that mass change lines are displaced horizontally along the temperature axis with the heating rate increase during the experiment. This could be also confirmed in Figures 1b-4b, since the sample mass change rate is presented as a peak on the related temperature. These findings could be explained by intensive sample mass loss during the thermochemical conversion in nitrogen atmosphere. Also, it could be shown that peaks of the conversion rate are obtained at different temperatures that are in the range between 550 K and 600 K for SCG and between 650 K and 700 K for WT, which is related to the composition of the volatile matter (VM) for examined samples. The composition of VM from SCG samples according to Figures 1 and 2 should consist of lighter hydrocarbons with lower evaporation temperatures as compared to VM from WT samples. The effect of particle size for this issue could be neglected.

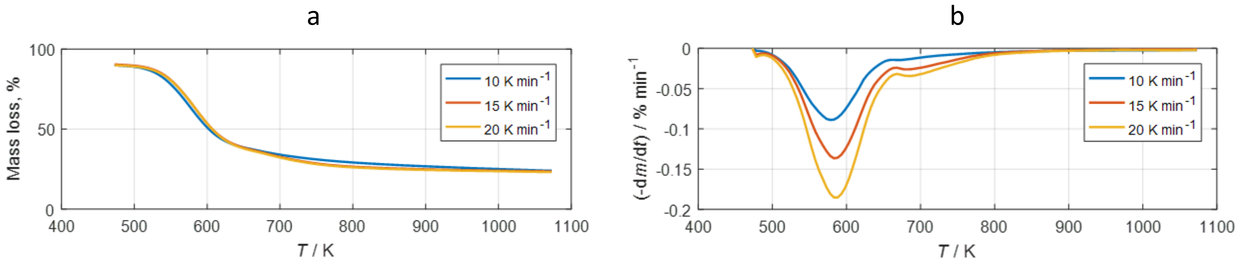

Figure 1. Mass change (a) and mass change rate (b) vs. *temperature for SCG (sample size x < 0.25 mm)*

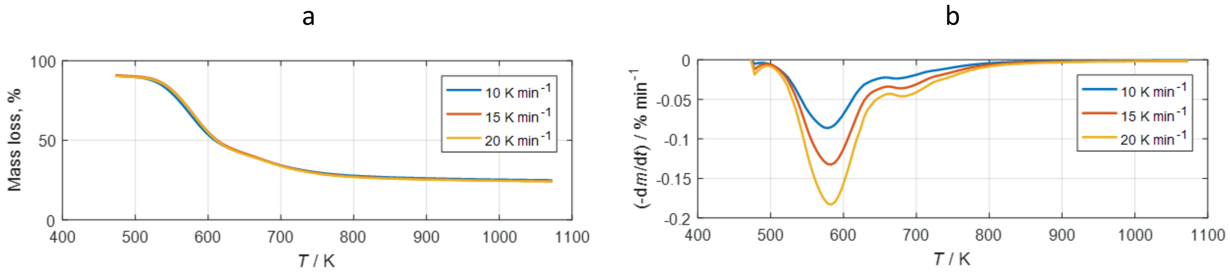

Figure 2. Mass change (a) and mass change rate (b) vs. *temperature for SCG (sample size 0.25 < x < 0. 5 mm)*

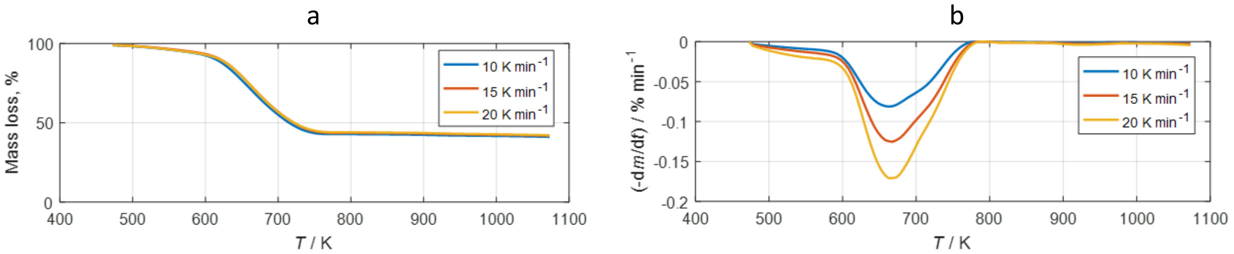

Figure 3. Mass change (a) and mass change rate (b) vs. *temperature for WT (sample size x < 0.25 mm)*

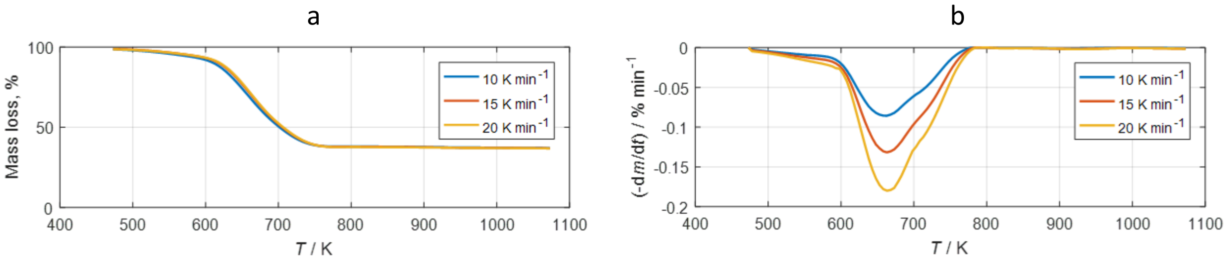

Figure 4. Mass change (a) and mass change rate (b) vs. *temperature for WT (sample size 0.25 < x < 0.5 mm)*

4.2. Results of the kinetic analysis

In this paper, SRF kinetic analysis was conducted using model-free methods: Friedman, KAS and FWO. Activation energy (E) was calculated from the slopes of regression lines, by plotting ln ($\mathrm{d}\alpha/\mathrm{d}t$) *vs.* $1/T$ for the Friedman, ln β *vs.* $1000/T$ for the FWO and ln (β/T^2) *vs.* $1000/T$ for the KAS method, for the given value of conversion (α = 0.1 - 0.8). The selected conversion value was chosen for all curves at three different heating rates. Coefficients of determination (R^2) were also calculated in order to determine which value of conversion provides the best statistical fit. Since the intercept of regression line was not performed in order to estimate the pre-exponential factor, the reaction model had to be assumed, at the selected order of reaction, $n = 2$.

As an example, diagrams of mean values of activation energy and pre-exponential factor, as well as plots for different conversion values are presented in Figures 5 to 7 for the WT sample size 0.25<x<0.5 mm.

Results presented in Figures 5, 6 and 7 are used for determination of the slope and intercept for obtained lines for different conversions α. Different kinetic methods are used then for calculation of kinetic parameters and their correlations with conversion are obtained (Figs 8 – 10).

Analysis of the obtained values of activation energy and frequency factor presented in Figures 8 – 10 obtained by different kinetic methods, shows a good agreement between these data and conversion α. These data could be used for determination of average values for kinetic parameters, which are important for mathematical modeling of thermochemical conversion process for selected materials and that will be the subject of further research of the authors.

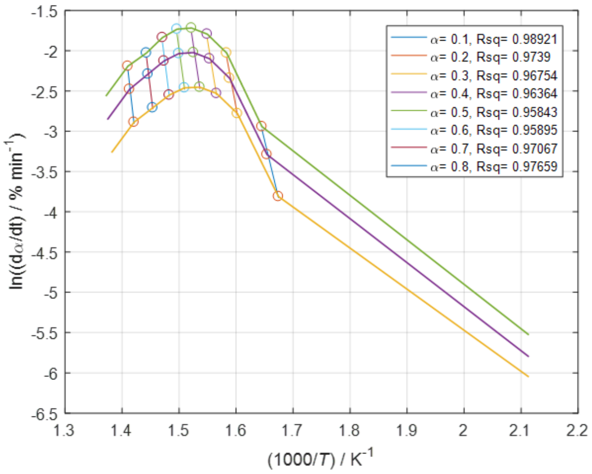

Figure 5. Friedman's plot of WT for different conversion values and the obtained coefficients of determination (WT, 0.25<x<0.5 mm)

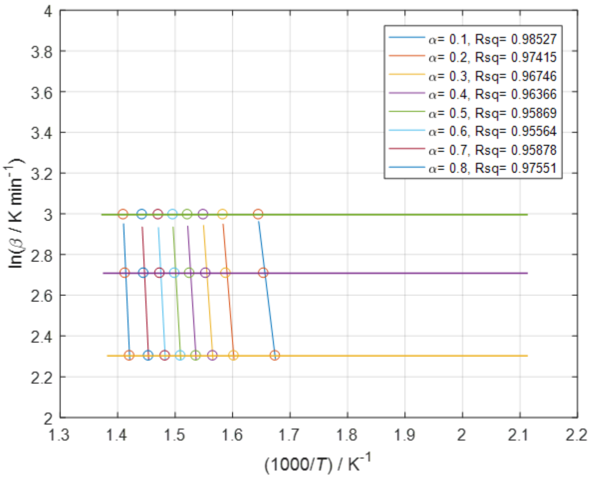

Figure 6. FWO plot of WT for different conversion values and the obtained coefficients of determination (WT, 0.25<x<0.5 mm)

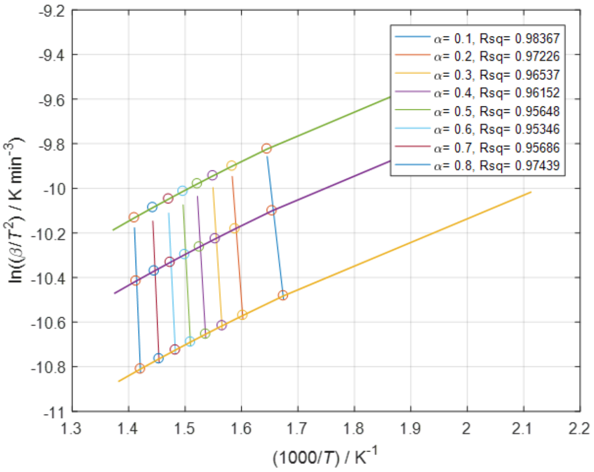

Figure 7. KAS plot of WT for different conversion values and the obtained coefficients of determination (WT, 0.25<x<0.5 mm)

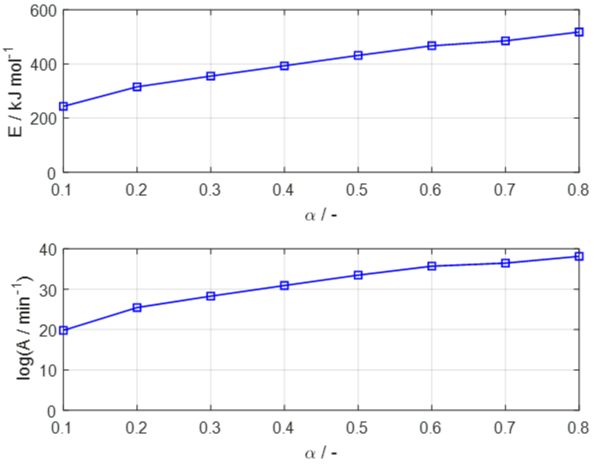

Figure 8. Activation energy and pre-exponential factor values for different conversion values of conversion – Friedman method

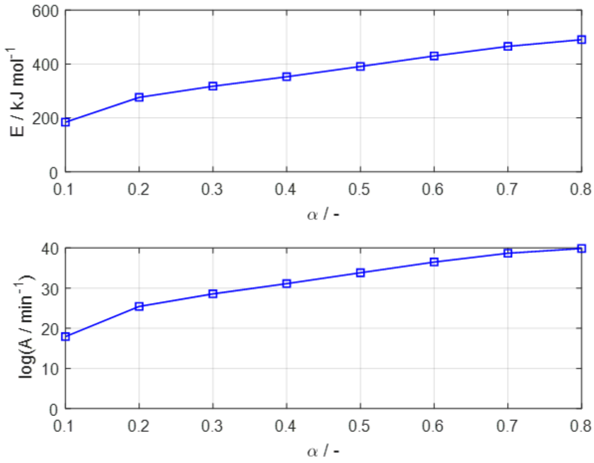

Figure 9. Activation energy and pre-exponential factor values for different conversion values – FWO method

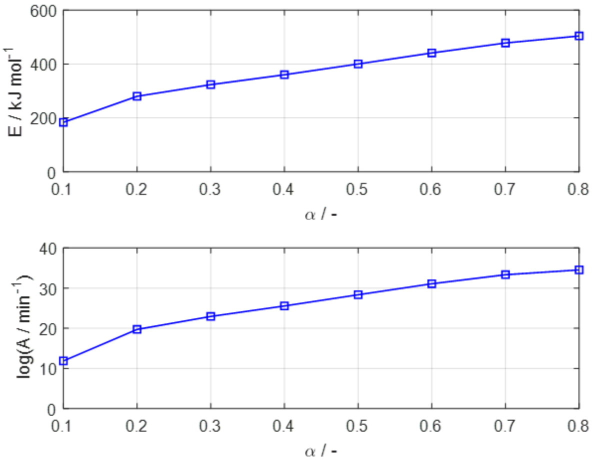

Figure 10. Activation energy and pre-exponential factor values for different conversion values – KAS method

In order to compare results obtained from described methods, activation energy and frequency factor mean values are grouped and presented in Figure 11:

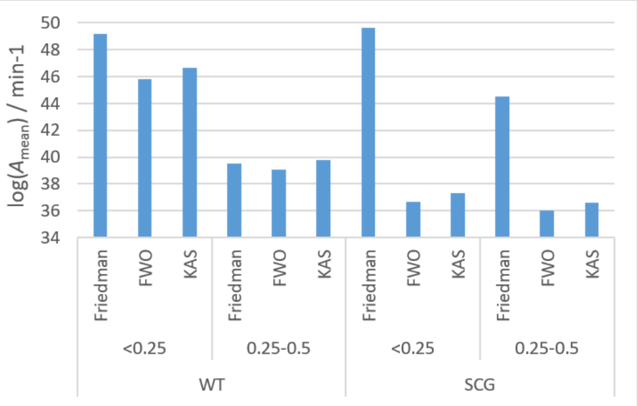

Figure 11. Comparison of pre-exponential factor values obtained by different isoconversional methods

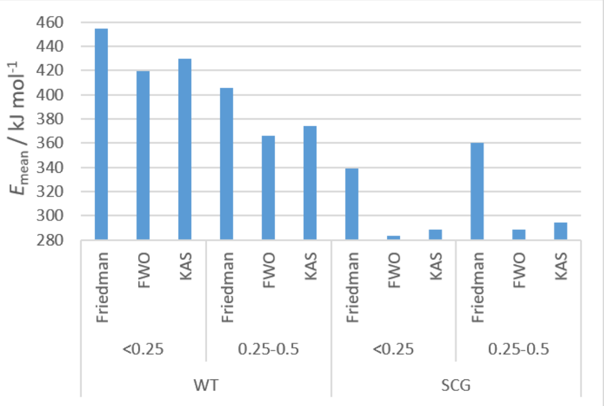

Figure 12. Comparison of activation energy values obtained by different isoconversional methods

From the Figures 11 and 12, it can be clearly seen that similar activation energy and pre-exponential values are obtained by FWO and KAS, while the values obtained by the Friedman method significantly differ. *E* values calculated using the Friedman's method for coffee samples are much higher than in the case of waste tires since in the former case *E* errors are more significant, considering the lower sensitivity of the instrument at measurements of the coffee samples due to the strong influence of thermal hysteresis when switching from 15 to 20 °C (lower sensitivity) which is not so significant in the case of waste tire samples. This is to be expected because the Friedman method is directly derived from the basic kinetic equation (eq. 11):

$$-\frac{\mathrm{d}m}{\mathrm{d}T}=k\cdot e^{-\frac{E}{RT}}\left(\frac{1-m_\tau}{m_0}\right)^n \tag{11}$$

Also, this issue could be explained by taking into account the correlation of thermal hysteresis with temperature variation during linear heating of the sample, which is associated with the coefficient C_1 ("thermal-lag" coefficient).

Even so, authors' opinion is that it is acceptable because same deviations are presented in other researches [1-14].

5. CONCLUSION

In this study, kinetic analysis of SRF pyrolysis was performed using TGA-DTG techniques and important kinetic information, such as the activation energy and pre-exponential factor were estimated using differential and integral isoconversional methods. Thermogravimetric analysis was performed in nitrogen atmosphere at different heating rates of 10, 15 and 20 K/min while pyrolysis process was analyzed in the temperature range from 200 °C up to 900 °C.

According to summary of presented results, the Friedman kinetic method provides higher values for kinetic parameters due to different approach for definition of conversion function $f(\alpha)$. Generally, the applied kinetic methods provided acceptable values of kinetic parameters which are comparable with literature data.

Further research will be focused on comparing the obtained kinetic parameters with other kinetic models/methods presented in the literature.

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SAŽETAK

Analiza kinetičkih parametara procesa pirolize čvrstih goriva iz otpada korišćenjem termogravimetrije

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(Naučni rad)

U Republici Srbiji postoji značajna količina otpada kafe i gume koja se može upotrebiti kao čvrsto gorivo iz otpada (engl. SRF – solid recovered fuel) i koristiti kao dodatno gorivo pri ko-sagorevanju sa ugljem ili biomasom u sektorima proizvodnje energije i industrije cementa. Značajne razlike između SRF i osnovnog goriva (uglja, biomase, itd.) su uzrok brojnih problema u projektovanju gorionika. Cilj ovog rada je bio da se odrede kinetički parametri termohemijske konverzije izabranog SRF koristeći simultanu termičku analizu (STA). Za eksperimentalna ispitivanja korišćeni su uzorci kafe i automobilskih guma. Termička analiza je obavljena u atmosferi azota sa tri brzine zagrevanja 10, 15 i 20 K/min, pri čemu je svaki uzorak zagrevan od sobne temperature do 900°C. U eksperimentima su korišćene dve granulacije uzorka x <0.25 mm i 0.25 < x <0.5 mm svakog od goriva, kako bi se dobili pouzdani podaci termičke gravimetrijske analize (TGA) za procenu kinetičkih parametara za pirolizu SRF. Eksperimentalni rezultati su korišćeni za određivanje predeksponencijalnog faktora i energije aktivacije prema izotermskim i neizotermskim metodama prikazanim u literaturi. Prikazana istraživanja su dala značajne podatke za otpad od kafe i autmobilskih guma koji se mogu koristiti u projektovanju peći za ko-sagorevanje čvrstog goriva iz otpada/uglja ili smeše biomase.

Ključne reči: simultana termička analiza, STA; termogravimetrijska analiza, TGA; kinetički parametri; čvrsta goriva iz otpada; piroliza .