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ATIK BİYOKÜTLE PELLETİNİN TERMOGRAVİMETRİK ANALİZİ VE YANMA KİNETİĞİ

(THERMOGRAVIMETRIK ANALYSIS OF WASTE BIOMASS PELLET AND COMBUSTION KINETICS)

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ÖZET

Bu çalışmada; tarımsal kalıntı saplarından (ayçiçeği, pirinç, mısır ve buğday) üretilen pelletin yanma karakteristiği ve kinetiği termogravimetrik analiz (TG) kullanılarak incelenmiştir. Pellet, bağlayıcı madde olarak *Euqhorbia denroides* (% 1) ile buğday (% 78) , mısır (% 13) , ayçiçeği (% 7) ve pirinç (% 1) karışımı ile oluşturulmaktadır. Ingraham-Marrier, Arrhenius ve Coats-Redfern izotermal olmayan kinetik modeller kinetik parametreleri hesaplamak için uygulanmıştır. Ingraham - Marrier modeli, Arrhenius ve Coats-Redfern modellerine göre tarımsal numunelerin yanma özelliklerini daha iyi bir şekilde tanımlamaktadır.

Anahtar Kelimeler: Tarımsal, Atık, Sap, Pellet, Yanma, Kinetik

ABSTRACT

In the present study, combustion properties and kinetics of agricultural residue stalks (sunflower, rice, corn, and wheat) and their respective pellet were investigated using thermogravimetric system (TG). Stalks pellet made from mixture of wheat (78%), corn (13 %), sunflower (7 %) and rice (1 %) with *Euqhorbia denroides* (1 %) as a binder. Ingraham–Marrier, Arrhenius, and Coats-Redfern non-isothermal kinetic models were applied to calculate the kinetic parameters. The Ingraham–Marrier model shows better prediction than the Arrhenius and Coats-Redfern models, and satisfactorily described the combustion of agricultural samples.

Keywords: *Agricultural, Residue, Stalks, Pellet, Combustion, Kinetics*

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1. INTRODUCTION

Current energy crisis in the world has increased and affected domestic energy consumption and many industries demands. State of affairs has driven many industries to utilize renewable resources for energy purpose. Biomass materials are considered to be one of the leading candidates for energy utilizations. In the future, biomass combustion will play an important role in energy production [1, 2].

Agriculture has always been one of the leading sectors in the Turkish economy, largely for natural reasons: the rich soil sources, biological diversity, good climate and geographical conditions. Although Turkey is an important producer of grains, with wheat yield of 1.95 tons per hectare, it is still lagging behind the EU-27 average yield of 5.66 tons per hectare [3]. The total recoverable bioenergy potential is estimated to be about 16.92 Mtoe. The estimate is based on the recoverable energy potential from main agricultural residues, livestock farming wastes, forestry and wood processing residues and municipal wastes as given in the literature. The biomass energy production for the year 2001 is 6.98 Mtoe [4].Turkey appears to be the one of the most efficient and effective country to obtained the energy from the agricultural residues.

Biomass materials with high energy potential include agricultural residues such as straw, bagasse, coffee husks and rice husks as well as residues from forest-related activities such as wood chips, sawdust and bark [5]. In addition to this, pellets, made from agricultural residues, are economic considerations for especially home owners and industrial users. Compared to traditional firewood, pellets provide possibilities for automation and optimization similar to oil, with high combustion efficiency and low combustion residues [6]. The global pellet market has grown quickly during the last decade and applications including combustion in grate furnace and gasification in fluidized bed furnace [7].

Characteristics of raw biomass and biomass pellets have obviously affected fuel quality. Due to differences in chemical and physical properties of the sources, replacing with traditional fossil fuels means that the combustion behavior is the main issue must be considered. It is also underline that the ignition of different biomass and pellets has a large impact on the emission levels [8].

Thermal analysis techniques are widely used in order to investigation of combustion behaviors of agricultural residue and pellets [9-13]. It were reported the effect of pelletizing conditions on combustion behavior of single wood pellet by using a laboratory scale furnace was equipped with an analytical balance enabling using it as a macro-TGA. It was shown that time required for single pellet combustion generally increased with pelletizing temperature. Pellets produced with wet biomass (moisture content: 12%) required longer combustion time than pellets produced with oven-dried biomass (moisture content: ~1%) [14]. Non-isothermal TG was applied to determine the combustion characteristic of six samples, namely wheat straw, rape straw, flax straw (leftover after scutching), pulp-mill lignin, garden peat, and hardwood charcoal. It was found that combustion of wheat straw showed a longer transition stage between volatilization and char burning [15].

The combustion of two kinds of biomass and sewage sludge was studied at different heating rates. The biomass fuels were wood biomass (pellets) and agriculture biomass (oat). KAS model was applied to calculate the activation energy (Ea) and Ea values for coal were in the range of 21.1-145.7 kJ mol^{-1}, for wood biomass 81.1–223.1 kJ mol^{-1} and for oat 11.9–282.5 $kJmol^{-1}$ [16].

2. EXPERIMENTAL

2.1. Materials and pellet preparation

Sunflower, rice, corn, and wheat stalks were used as an agricultural material in the present study. We chose these waste, because there are huge amounts of agricultural waste are easy to be obtained in Uzunköprü/ Edirne in Marmara Region in Turkey. Natural dried agricultural stalks were grounded and sieved to <250μm standard sieves (as determined by the American Society for Testing and Materials) before the combustion analysis. Elemental analyses of the agricultural stalks were conducted in accordance with ASTM D3172-07a using a LECO CHN-600 carbon-hydrogen-nitrogen analyzer and the total sulphur content was determined by a LECO SC-132 sulfur analyzer (Table 1).

Table 1. Elemental analyses of the agriculture stalks and pellet

Sample	C*/%	H*/%	N*/%	S*/%
Wheat	42.80	5.55	0.45	0.19
Rice	37.47	4.97	0.81	0.12
Corn	43.17	5.55	1.63	0.11
Sunflower	43.55	5.63	0.29	0.03
Pellet	42.85	5.55	0.60	0.17

* on the dry basis

The proximate analyses of the samples were performed in accordance with ASTM standard (ASTM E1131–03) and the calorific value was determined in accordance with ASTM D 5865-04 by a bomb calorimeter (IKA-Calorimeter C400) (Table 2). An average on three samples was taken for all mentioned analyses.

Table 2. Proximate analyses of the agriculture stalks and pellet

Sample	Moisture/%	Ash/%	Volatile mater /%	Fixed carbon/%	Calorific values*/calgr^{-1}
Sunflower	8.42	2.06	87.36	2.16	3329
Rice	5.90	11.38	72.29	10.43	3200
Corn	8.80	8.78	78.76	3.66	3640
Wheat	6.13	3.20	86.87	3.80	3527
Pellet	6.26	6.28	68.56	18.90	3758

* on dry basis

Pelleting experiments were conducted by using an extruder and cylindrical shape was obtained with 6 cm diameter and 31 cm length. Stalks pellet made from mixture of wheat (78%), corn (13 %), sunflower (7 %) and rice (1 %) with *Euqhorbia denroides* (1 %) as a binder. Before pelleting, agricultural residues were grinded at room temperature for 2 min. and all agricultural residues were mixed in the presence of binder according to the formula After the pressing of mixture of agricultural residues in the extruder, pellets were obtained

(Figure 1). Elemental and proximate analyses of the agriculture stalks pellet were determined by same methods describe above (Table 1-2).

Figure 1. Photo of agricultural pellet used in the study

2.2. Combustion analysis

Combustion of agricultural stalks and their respective pellet were carried out using the Perkin Elmer Diamond DTA/TG instrument, which was calibrated using of the melting points of indium (T_m=156.6°C) and tin (T_m=231.9°C) under the same conditions as the sample. The analyses were carried out at 10 °C/min heating rate in atmosphere of O_2 that had a constant flow rate of 100 ml/min. The samples (~10 mg) were allowed to settle in standard platinum crucibles and heated up to 700 °C. TG profiles are given in Figure 2 and inset shown the DTG profiles.

2.3 Combustion kinetics

In the present study, combustion reaction of the agricultural samples can be defined as below:

Agricultural stalk / pellet $_{(s)}$ $\rightarrow$ Volatiles $_{(g)}$ + Ash $_{(s)}$ (1)

Kinetic analysis of combustion reactions of agricultural stalks and their respective pellet were investigated by using mathematical equations of Ingraham-Marier, Arrhenius and Coats-Redfern non-isothermal kinetic models.

In the Ingraham- Marrier method, reaction order is assumed to 1. The calculation of kinetic parameters was made based on Eq. 2. Log (dw/dT) values were plotted agasit to the 1/T to obtain kinetic curve and apparent E_A is calculated from the slope and k_0 can be determined from the intercept [17].

$$log\frac{dW}{dT} = logT + log\ a + logk_o - \frac{E_A}{2.303RT} \quad (2)$$

In the Arrhenius method (Eq.3), the rate of mass loss of the total sample depends only on the rate constant, the mass of sample remaining and the temperature and reaction order is assumed to 1. Log [(dw/dT).(1/W)] values were plotted against to the 1/T and apparent E_A is calculated from the slope and k_0 can be determined from the intercept [18].

$$\log\left[\left(\frac{dW}{dt}\right)\times\left(\frac{1}{W}\right)\right] = \log k_0 - \frac{E_A}{2.303RT} \tag{3}$$

According to the Coats-Redfern method, values of [log (-log(1-α)/T^2)] versus 1/T was plotted. The slope of the line was used to calculate E_A and also k_0 was determined from the intercept of the line. To calculate the kinetic parameters, thermal dehydration reaction mechanism is assumed first order (n=1).

$$\log\left[\frac{-\log(1-\alpha)}{T^2}\right] = \log\frac{k_0 R}{\beta E_a}\left[1-\frac{2RT}{E}\right] - \frac{E_A}{2.303RT} \tag{4}$$

2.4 Statistical analysis

The statistical analysis of experimental data was determined using Statistica 6.0 software (Statsoft Inc., Tulsa, OK), which is based on the Levenberg–Marquardt algorithm. The three criteria of statistical analysis have been used to evaluate the adjustment of the experimental data to the different models: the coefficient of determination (R^2), reduced chi-square (χ^2) and root-mean-square error (RMSE). The best model describing the combustion characteristics of samples was chosen as the one with the highest R^2, the least χ^2 and RMSE. These parameters can be calculated as:

$$\chi^2 = \frac{\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{pre,i}\right)^2}{N-z} \tag{5}$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2\right]^{1/2} \tag{6}$$

where $MR_{exp,i}$ and $MR_{pre,i}$ are the experimental and predicted dimensionless MR, respectively, N is the number of data values, and z is the number of constants of the models.

3. RESULT AND DISCUSSION

3.1 Combustion characteristics

TG and DTG profiles of combustion of the their respective pellet are given in Figure 2 and Table 3 gives data obtained through interpretation of these profiles.

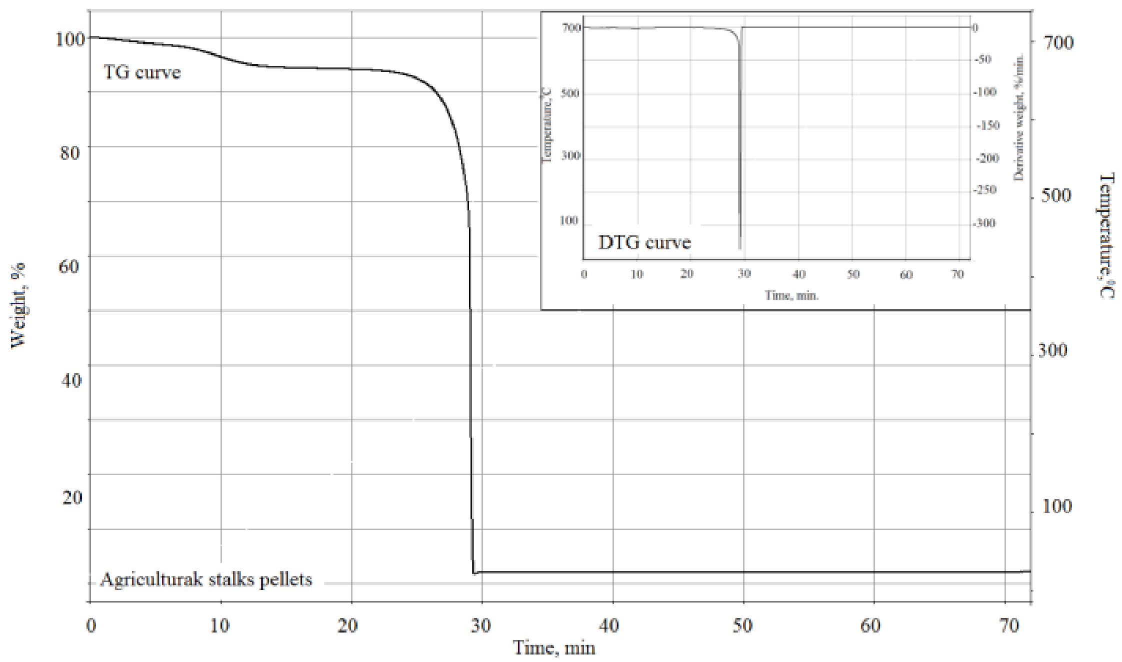


Figure 2. TG profile of agriculture stalk pellet. The inset showed the DTG curves

Considering each agricultural stalks and their respective pellet, the profile of TG and DTG curves are exhibit a similar combustion behavior. It can be seen that the combustion of agricultural stalks and pellet can be divided in to 2 steps such as initial (Step I) and main (Step 2). Step 1 account for moisture evaporation and the step 2 is due to oxidative degradation. For all stalks and pellet the main stage started quickly after moisture evaporation. Moisture evaporation was occurred at the step 1 in the temperature ranges 36.63-128.63 °C for sunflower stalk, 43.15-175.28 °C for rice, 39.67-134.93 °C for corn, 39.36-172.32 °C for wheat. No significant difference in the temperature range in the initial step for the stalks. In addition to this, demoisturization of agricultural pellet was occurred at 27.49-164.43 °C temperature range that was lower than the stalks. After further heating the step 2 was started and continues up to about 500 °C. This region was associated with devolatilization of cellulose components and their ignition [19]. Overall observed weight losses were 86.51 %, 82.42 %, 82.56 %, 88.96 %, and 91.32 % for sunflower, rice, corn, wheat, and pellet, respectively. It is also apparent that minimum ash amount was observed after the combustion of agricultural pellet compared with the stalks. On the DTG curves the temperatures (T_m, °C) at which maximum rates of weight loss (R_m, %min^{-1}) were determined. DTG curves shows two peaks during the combustion of agricultural stalks and pellet associated with moisture evaporation and ignition. R_m values of moisture evaporation reaction at peak temperatures of 61.28 °C, 63.74 °C, 70.54 °C, 67.07 °C and 53.79 °C were 1.50 %min^{-1}, 1.34 %min^{-1}, 1.23 %min^{-1}, 1.11 %min^{-1}, and 0.80 %min^{-1} for sunflower, rice, corn, wheat, and pellet, respectively. In the step 2 it was also found that R_m values at peak temperatures of 298.20 °C, 307.00 °C, 301.40 °C, 323.44 °C and 317.95 °C were 179.65 %min^{-1} , 252.02 %min^{-1} , 172.30 %min^{-1} , 442.06 %min^{-1}, and 336.05 %min^{-1} for same sample sequence. Under the oxidative environment, ignition leads to the more rapid weight loss. It is well know that the ignition characteristic is based on physical, structural and elemental characteristics of biomass components.

Table 3. Thermogravimetirk analysis results of agriculture stalks and pellet

Stalk	Step	T_i (°C)	T_f (°C)	m (%)
Sunflower	I	36.63	128.63	7.79
	II	191.56	415.32	78.72
Rice	I	43.15	175.28	6.94
	II	195.16	434.14	75.48
Corn	I	39.67	134.93	8.34
	II	177.19	347.92	74.22
Wheat	I	39.36	172.32	5.94
	II	186.16	426.48	83.02
Pellet	I	27.49	97.03	4.65
	II	164.43	498.03	86.67

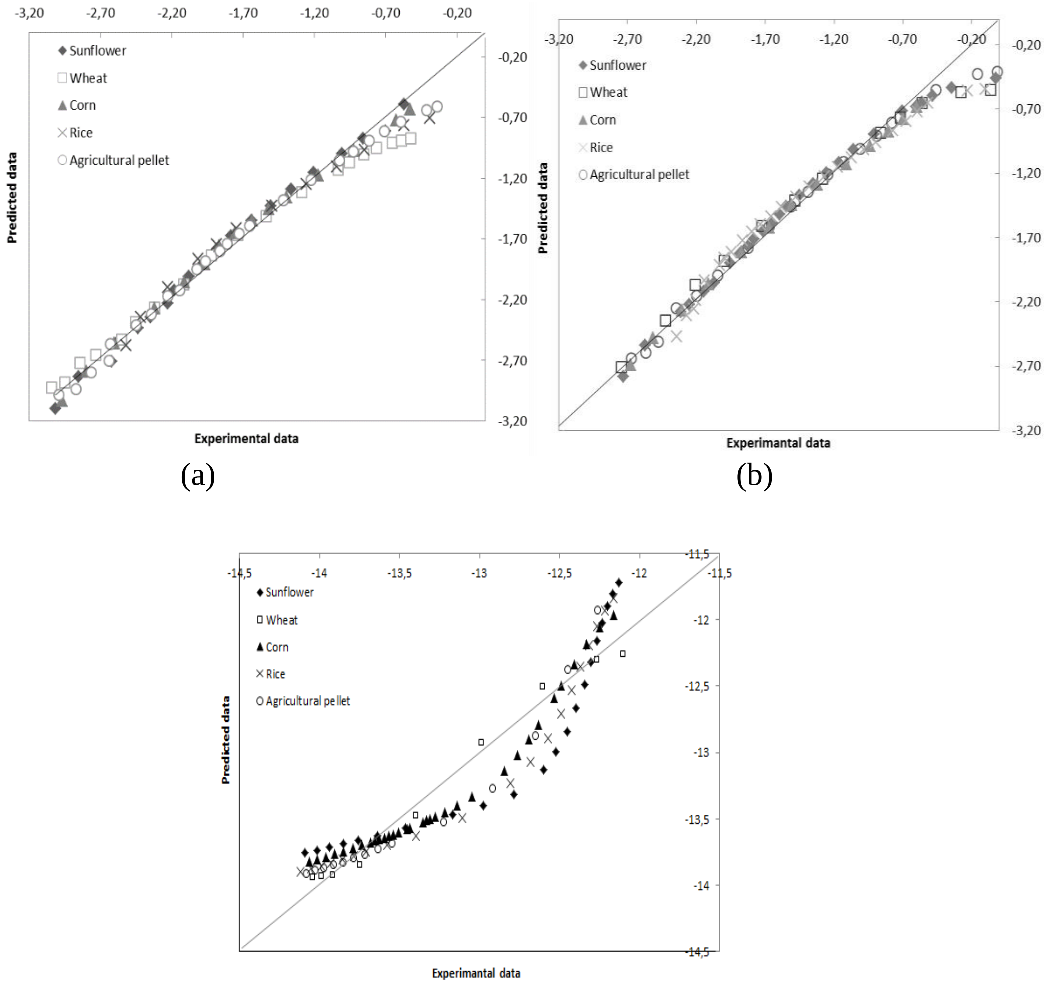
3.2 Combustion kinetics

Kinetic calculations were performed to the step 2 associated with the combustion reaction. The data obtained using Ingraham–Marrier, Arrhenius, and Coats-Redfern non-isothermal kinetic models are given in Table 5 with curve fitting criteria values for models. Apparent E_a and k_o were calculated assuming the reaction degree to be 1. Apparent E_a calculated with this region from Arrhenius model were 112.22 kJmol^{-1}, 136.93 kJmol^{-1}, 127.49 kJmol^{-1}, 113.88 kJmol^{-1} and 124.35 kJmol^{-1} for sunflower, rice, corn, wheat, and pellet, respectively. In addition to this, 104.23 kJmol^{-1}, 127.58 kJmol^{-1}, 131.73 kJmol^{-1}, 102.74 kJmol^{-1} and 115.30 kJmol^{-1} values for same sample sequence were determined by applying Ingraham & Marier model. The calculated kinetic parameters were varied with method used and Ingraham & Marier model yielded the lowest apparent E_a for all samples. Apparent E_a values were fairly close agreement with literature data reported on apparent E_a of biomasses combustion as wheat straw (111 kJmol^{-1}) [20], bagasse (127.49 kJmol^{-1}) [21], cotton stalk (119.90 kJmol^{-1}) [22].

Weight loss data obtained at oxidative atmosphere for sunflower, rice, corn, wheat, and pellet were used and two non-isothermal kinetic models (Ingraham–Marrier, Arrhenius and Coats-Redfern) were used to calculate the kinetic parameters (E_a and k_o). Nonlinear regression was used to obtain each parameter value of every model. The statistical results from models such as coefficient of determination (R^2) and reduced chi-square (χ^2) values are summarized in Table 4. The best model describing the combustion characteristics of agricultural samples was chosen as the one with the highest R^2 values and the lowest χ^2 and RMSE values. In kinetic calculation, if the R^2 values for the models were greater than the acceptable R^2 value of 0.90, it is indicated that a good fit of experimental to predicted data. In the present study, R^2, χ^2, and RMSE values were changed between 0.9408-0.9904, 0.003512-0.018790, and 0.05907-0.212592, respectively. It is found that Ingraham–Marrier model gives the highest values of R_2 and the lowest values of χ^2 for all the samples. Also, lower RMSE values were obtained with the application of Ingraham–Marrier model. Therefore, the Ingraham–Marrier model shows better prediction than the Arrhenius and Coats-Redfern models, and satisfactorily described the combustion of agricultural samples. Fig. 3 shows the comparison of experimental data with those predicted with the Ingraham–Marrier, Arrhenius and Coats-Redfern models.

Table 4. Curve fitting criteria values and constants for models and parameters for agriculture stalks and pellet

Model	**Samples**	**Constants (k_0, min^{-1} E_A, $kJmol^{-1}$)**	**R^2**	**RMSE**	**χ^2**
Arrhenius	Pellet	Log k_0 = 10 E_A = 112.22	0.9884	0.069897	0.004928
	Sunflower	Log k_0 = 12.30 E_A = 136.93	0.9758	0.107127	0.011541
	Wheat	Log k_0 = 11.60 E_A = 127.49	0.9408	0.212592	0.045450
	Corn	Log k_0 = 10.20 E_a= 113.88	0.9869	0.079319	0.006355
	Rice	Log k_0 =10.90 E_A = 124.35	0.9558	0.136544	0.018790
Ingraham - Marier	Pellet	Log k_0 =9.40 E_A=104.23	0.9904	0.05907	0.003512
	Sunflower	Log k_0 = 11.60 E_A= 127.58	0.9814	0.08702	0.007606
	Wheat	Log k_0 =12.30 E_A=131.73	0.9710	0.1049061	0.011106
	Corn	Log k_0=9.20 E_a=102.74	0.9884	0.061247	0.003770
	Rice	Log k_0 =10.20 E_A=115.30	0.9609	0.118002	0.014046
Coats - Redfern	Pellet	Log k_0 =-6.09 E_A=35.18	0.9080	0.155030	0.025246
	Sunflower	Log k_0 = -2.61 E_a= 50.66	0.9060	0.276584	0.080866
	Wheat	Log k_0 =-6.67 E_A=32.35	0.9844	0.082700	0.007816
	Corn	Log k_0=-4.56 E_A=41.43	0.9236	0.149990	0.023101
	Rice	Log k_0 =-3.54 E_A=47.39	0.9042	0.190273	0.039182



(c)

Figure 3. Distribution of experimental and predicted weight lost data for non-isothermal kinetic models; (a) Arrhenius, (b) Ingraham- Marrier, (c) Coats - Redfern

4. CONCLUSION

In this study, combustion behavior of several types of agricultural stalks (sunflower, rice, corn, and wheat) and their respective pellet studied using thermogravimetric system (TG) under oxidative atmosphere. Ingraham - Marier and Arrhenius non-isothermal kinetic models were applied to calculate the devolatilization kinetic parameters. The following points result from this study:

1. There are no significant differences in the combustion characteristic for the same type agricultural stalks include lignin, cellulose, and hemicellulose. Combustion of agricultural stalks and pellet can be divided in to 2 steps such as initial (Step I) and main (Step 2). Step 1 account for moisture evaporation and the step 2 is due to oxidative degradation.

2. In addition to this, demoisturization of agricultural pellet was occurred at 27.49-164.43 °C temperature range that was lower than the stalks.

3. After further heating the step 2 was started and continues up to about 500 °C. This region was associated with devolatilization of cellulose components and their ignition.

4. The Ingraham–Marrier model shows better prediction than the Arrhenius and Coats-Redfern models, and satisfactorily described the combustion of agricultural samples.
5. Apparent E_a calculated with this region from 104.23 $kJmol^{-1}$, 127.58 $kJmol^{-1}$, 131.73 $kJmol^{-1}$, 102.74 $kJmol^{-1}$ and 115.30 $kJmol^{-1}$ values sunflower, rice, corn, wheat, and pellet, respectively. Apparent E_a values were fairly close agreement with literature data reported on apparent E_a of biomasses combustion.

NOMENCLATURE

W	Weight
T	Temperature
E_A	Activation Energy
k_0	Arrhenius costant
α	Decompositon fraction
R	Gas Constant
RMSE	Root mean square error
χ^2	Reduced chi-square
R^2	Coefficient of determination
$MR_{exp,i}$	Experimental dimensionless moisture ratios
$MR_{pre,i}$	Predicted dimensionless moisture ratios
N	Number of observations

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