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Thermal Behavior and the Role of Minerals of Agricultural Waste/Lignite Blends on Combustion Performance and Slagging/Fouling Propensity

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***Abstract*— Thermal behavior in air of agricultural waste/lignite blends was studied in terms of ignition, reactivity and burnout characteristics. The role of minerals on the burning behavior of the mixtures and on slagging/fouling propensities was investigated by carrying out chemical, mineralogical and fusibility analyses of ashes, along with co-combustion experiments. Lignite/corn residue blends presented a high degree of additivity, while lignite/grape hull blends exhibited some synergy. Potassium of biomass materials acted as catalyst during combustion. Co-combustion of lignite with biomass fuels after reduction of ash improved its reactivity. Initial deformation of raw ash materials was low for most combustion processes. Upon leaching biomass fuels with water, the slagging/fouling propensity of the blends was lowered.**

***Keywords*— co-combustion; lignite; agricultural wastes; fouling**

I. INTRODUCTION

Co-combustion of coal with waste materials is receiving a lot of attention in recent years for sustainable heat and power production worldwide, due to declining of fossil fuel reserves and the environmental constraints associated with them. The advantages of this technology are both environmental and economic. The joint combustion of coal with biomass residues reduces greenhouse gas emissions, as well as the large volume of wastes for landfilling [1-3]. Furthermore, it allows for energy recovery, by replacing part of conventional fuels in existing infrastructures, thus avoiding large investments for new biomass plants [2].

Agricultural wastes, being abundantly found in most countries at low cost, are an attractive coal replacement option, in view of the limited availability of high quality woody materials with increased price. Also, their exploitation for energy generation meets the European Union Agricultural and Renewables Policy [2,4]. On the other hand, these wastes having different properties than coals, such as higher volatiles content, lower density and calorific value and greater concentration of alkali, could affect negatively fuel feeding, flame stability, ash fouling and consequently plant efficiency and electricity cost [2,5-7]. Moreover, possible mutual interactions between component fuels during co-firing may or may not be beneficial [5,6,8-11]. Therefore, the study of the thermal behaviour of coal/biomass blends during combustion is critical, in order to avoid fuel combinations with unwanted properties.

Co-combustion of selected lignocellulosic materials and municipal solid wastes has been extensively studied [12-15]. Synergy and slagging phenomena have been reported [5,8,10,15] and many different results have been obtained, due to the great diversity between coal and biomass properties. The majority of these investigations employed high rank coals [6,13-15] and only a few focused on low rank coals [8,10]. However, for many countries around the world, including Greece, lignite, accounting for about 40% of global coal reserves [16] is the indigenous fuel and thus very important for national economies. Additionally, the effect of chemistry and mineralogy of inorganic constituents of coal/biomass mixtures on the fusibility behaviour of ashes during combustion, determining slagging and fouling phenomena in boilers, has been scarcely addressed [17,18].

Based on the above, present work aimed to study the thermal behaviour in air of some agricultural waste/lignite blends in terms of ignition, reactivity, burnout characteristics and possible mutual interactions. Also, the work aimed to investigate the role of minerals on the combustion performance of the blends and slagging/fouling propensities. Chemical, mineralogical and fusibility analyses of ashes were carried out, along with the application of a simple ash reduction method and thermogravimetric analysis experiments. The results are expected to offer useful information for the co-firing of these fuels in lignite operated power plants.

II. EXPERIMENTAL

The lignite selected for the combustion tests was provided from the mines of Western Macedonia in Greece. Grape hulls and curls (GHC) were collected from a spirit making factory in the region of Apokorona in Crete, while corn residues (CR) were obtained from a field in Central Greece. After air drying and riffling, the lignite was ground in a jaw crusher and a ball mill, whereas the residues in a cutting mill. All materials were sieved to a particle size ≤ 200μm, in order to assure that the experiments would be carried out in a kinetic regime. Also, each biomass material was blended with the lignite at a ratio 30:70, which is typical for co-firing power plants in Europe [3].

A water leaching technique was applied to biomass samples, in order to investigate its effect on the combustion performance of the fuels and their mixtures, as well as on the fusion behaviour of ashes. De-ionised water was added to each sample at a ratio 120 mL/1g and the slurry was agitated for 2 hrs at 80°C. Solid materials were filtered and dried in the oven at 110°C.

For the characterization of fuels in terms of proximate analysis, ultimate analysis and calorific value, ASTM standards (D5142, D5373, D5865) and European CEN/TC335 standards were adopted for the lignite and biomasses, respectively.

Chemical analysis of ashes was conducted by an X-ray fluorescence spectrometer (XRF), type S2 Ranger/EDS, while mineralogical analysis by an X-ray diffractometer (XRD), type D8 Advance, both provided by Bruker AXS company. Fusibility analysis was performed by a heating microscope equipped with a high definition camera, model Leitz Wetzlar EM201 of Hesse Instruments. Ash samples were heated at a rate of 5°C/min in an oxidizing atmosphere up to 1550°C and the characteristic fusion temperatures were recorded.

Combustion experiments of raw fuels, leached biomass fuels and lignite/biomass blends were carried out in a differential thermobalance analyzer, type TGA-6/DTG of Perkin Elmer. The system records the sample weight loss and rate of weight loss (DTG curve) under dynamic conditions, as a function of temperature and time up to 1000ºC (sensitivity <5 μg, accuracy 0.2% and temperature precision ±2ºC). Pure nitrogen/air with a flow rate of 35 mL/min was used for the determination of volatiles and combustion parameters, respectively. The heating rate was 10^{o}C/min. Reproducibility of data, expressed as the relative standard deviation (RSD), was found to be very good. The thermograms were processed to determine characteristic parameters. Fuel reactivity was expressed as the ratio of peak rate to peak combustion temperature [19].

III. RESULTS AND DISCUSSION

A. Proximate and ultimate analysis of fuels

Proximate and ultimate analysis results of raw and pre-treated materials are represented in Table I. Both biomasses were richer in volatiles in comparison to lignite and had lower ash content. Upon leaching with water the amount of volatiles was increased, while that of ash was reduced by 50 to 70%. The higher concentrations of carbon and hydrogen of GHC biomass in conjunction with its lower ash content, with respect to lignite, resulted in a higher calorific value. The concentration of sulfur in all samples was very low, revealing negligible emissions of SO_x during combustion. However, the content of nitrogen of lignite and GHC was considerable, which could produce toxic NO_x emissions during the co-combustion process. The influence of leaching on the elemental composition of corn residues and grape hulls is reflected by the H/C and O/C molar ratios, which were lowered after the pre-treatment, leading to a rise in heating value. Also, it is interesting to note that for GHC these ratios attained similar values to those corresponding to lignite after the leaching process. Moreover, Table I shows that no nitrogen or sulfur compounds were dissolved in water, when grape hulls were leached.

TABLE I. PROXIMATE AND ULTIMATE ANALYSES AND CALORIFIC VALUE OF THE FUELS (% DRY WEIGHT)

Sample	***Volatile matter***	***Fixed carbon***	***Ash***	***C***	***H***	***N***	***O***	***S***	***H/C***	***O/C***	***GCV^{a} (MJ/kg)***
Corn residues (CR)	80.1	18.2	1.7	44.0	5.9	-	48.4	-	1.60	0.82	15.8
Corn residues leached (CR_l)	90.8	8.4	0.8	44.6	5.9	-	48.7	-	1.58	0.81	16.0
Grape hulls & curls (GHC)	71.5	14.3	14.2	49.4	6.5	2.4	27.1	0.4	1.57	0.41	20.7
Grape hulls & curls leached (GHC_l)	72.4	23.7	3.9	59.6	6.9	3.2	25.9	0.5	1.40	0.32	25.4
Lignite (LIG)	57.2	17.5	25.3	46.0	5.7	2.2	20.3	0.5	1.48	0.33	19.3

[a] Gross calorific value

B. *Combustion behavior of raw and leached fuels*

A comparison between the burning profiles of raw biomass fuels and those leached with water is made in Fig. 1 and 2. For both raw corn residues and grape hulls thermal decomposition started around 200°C. Major weight loss occurred between 220 and 350°C, presenting a high temperature peak assigned to cellulose, combined with a lower peak on the left, appearing as a shoulder to it, attributed to hemicellulose [5,11]. In the case of grape hulls sample, the contribution of hemicellulose was clearer. According to the pyrolysis profiles (not shown in the graphs due to similarity), volatiles were released and burned in this temperature region. Combustion of secondary gases and char were burnt with a lower rate, corresponding to lignin fraction, at temperatures 350-550°C.

When mineral matter was partially removed by washing the two biomass materials, Fig. 1 and 2 clearly illustrate that the whole DTG profile was shifted to higher temperature values (30 to 40°C), while the combustion rate was significantly lowered (about 1.5 times). This increased reactivity of the samples in air induced by the minerals is owned to the catalytic effect of potassium, which was abundant in raw biomass fuels and was reduced by 59 to 72% after washing, as it will be shown below. Similar results have been reported by several investigators, including the authors [11,20,21].

Table II presents the combustion parameters of raw and leached biomass fuels, as determined by processing of the TGA curves. Ignition temperature T_i (calculated as described in a previous work by the authors [5]) is known to be influenced by the volatiles content and energy, the H/C ratio of the fuel and its impurities. As can be observed from the table, grape hulls ignited earlier than corn residues, threshold R_{max} was about 3-fold lower, while burnout temperature T_b and time t_b were higher in comparison to corn residues. Additionally, leached samples followed the same trend. In terms of reactivity R_f, the fuels followed the order: CR > CR$_l$ > GHC > GHC$_l$.

TABLE II CHARACTERISTIC COMBUSTION PARAMETERS OF RAW AND LEACHED FUELS

Sample	***T_i* (°C)**	***T_{max}* (°C)**	***R_{max}* (10^2/min)**	***R_f* (10^2/min°K)**	***T_b* (°C)**	***t_b* (min)**
CR	200	295	26.1	18.3	500	39.0
CR$_l$	230	333	16.1	12.0	536	42.6
GHC	176	289	9.0	6.6	750	64.0
GHC$_l$	194	321	5.9	3.9	685	57.5
LIG	210	363	4.2	2.7	730	62.0

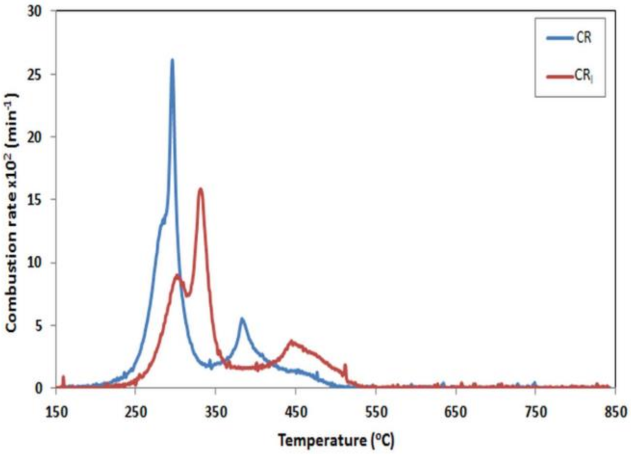


Fig. 1. DTG burning profiles of corn residues(RSD=0.6-0.7%)

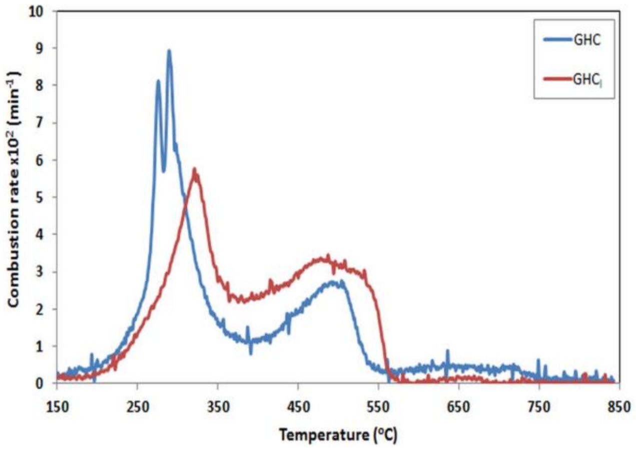


Fig. 2. DTG burning profiles of grape hulls & curls(RSD=0.7-0.9%)

C. Combustion behavior of raw and leached fuel blends

Fig. 3 and 4 compare the burning profiles of lignite/biomass blends. As can be seen, the major weight loss of lignite occurred between 220 and 480°C, corresponding to combustion of volatiles and char material, with a rate 2 to 6 times lower than that of biomass fuels ($R_{max,}$ Table II). The lower reactive char component continued to burn up to ~600°C. The small peak observed at ~710°C represents the decomposition of calcium carbonate of lignite, as it will be shown below.

In the case of lignite/corn residues mixture, Fig. 3 shows that ignition temperature was 205°C, i.e. coincided with the ignition temperature of corn residues, while peak temperatures of both lower and higher temperature regimes were practically the same as those of raw lignite. In other words, component fuels seemed to burn independently. On the other hand, for lignite/grape hulls mixture, although the contribution of each component fuel is evident, peak position was shifted to ~20°C higher temperature value than that of raw lignite and its height was lower. Consequently, it is likely that some mutual interaction occurred between lignite and grape hulls when blended. These results are summarized in Table III. Also, it can be noticed that when corn residues were mixed with the lignite, the reaction rate was increased and the burnout temperature and time were decreased, thus improving the reactivity and combustion efficiency of the lignite. However, in the case of lignite/grape hulls mixture reactivity was reduced with respect to lignite.

TABLE III. CHARACTERISTIC COMBUSTION PARAMETERS OF RAW AND LEACHED FUEL MIXTURES

Sample	***T_i* (°C)**	***T_{max}* (°C)**	***R_{max}* (10^2/min)**	***R_f* (10^2/min°K)**	***T_b* (°C)**	***t_b* (min)**
LIG/CR	200	363	5.7	4.0	710	60.0
LIG/CR$_l$	204	349	9.6	6.6	715	60.5
LIG/GHC	201	382	3.8	2.2	720	61.0
LIG/GHC$_l$	209	386	4.1	2.4	724	61.4
LIG	210	363	4.2	2.7	730	62.0

Upon leaching biomass materials with water, Fig. 4a indicates that for lignite/corn residues leached combustion rate was enhanced, due to the increased calorific value of the blend after reduction of ash and its maximum was displayed at a lower temperature. Furthermore, for lignite/grape hulls leached mixture, the combustion rate was also enhanced in comparison to raw mixture, as Fig. 4b shows. From Table III it can be seen that co-combustion of lignite with the leached biomass materials improved its reactivity R_f, which followed the order: LIG/CR$_l$ > LIG/CR > LIG/GHC$_l$ > LIG/GHC.

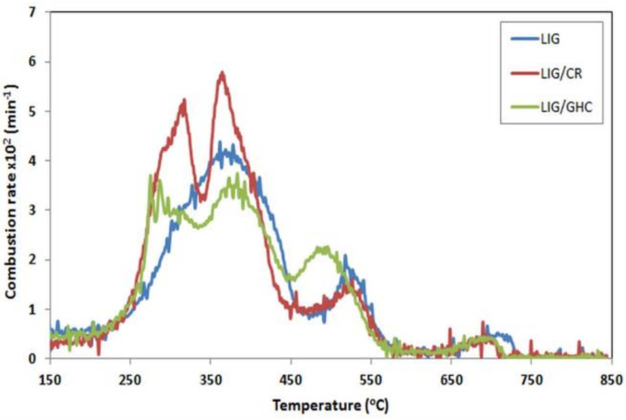


Fig. 3. DTG burning profiles of lignite/biomass mixtures (RSD=1.2-2%)

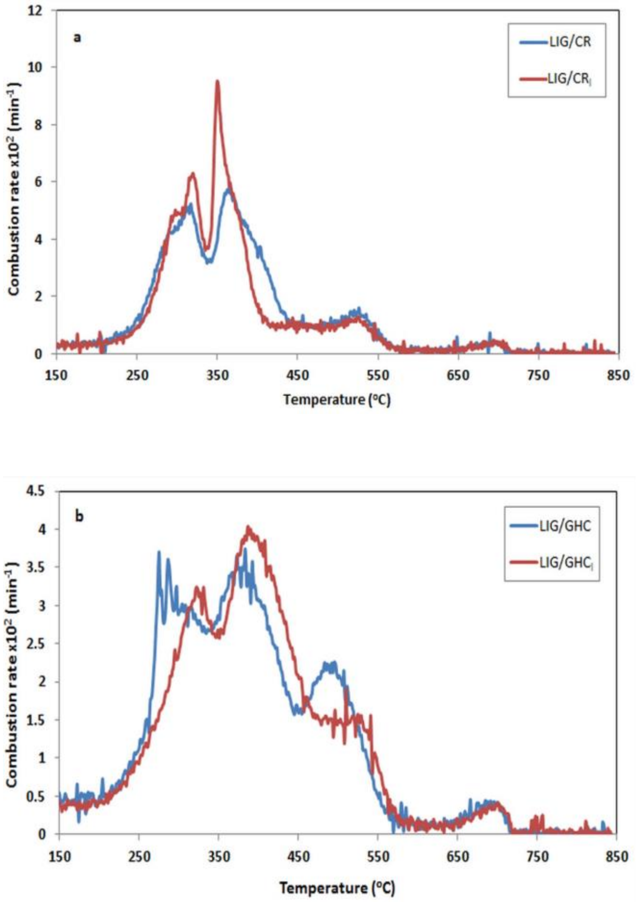


Fig. 4. DTG burning profiles of (a) lignite/corn residues and (b) lignite/grape hulls mixtures

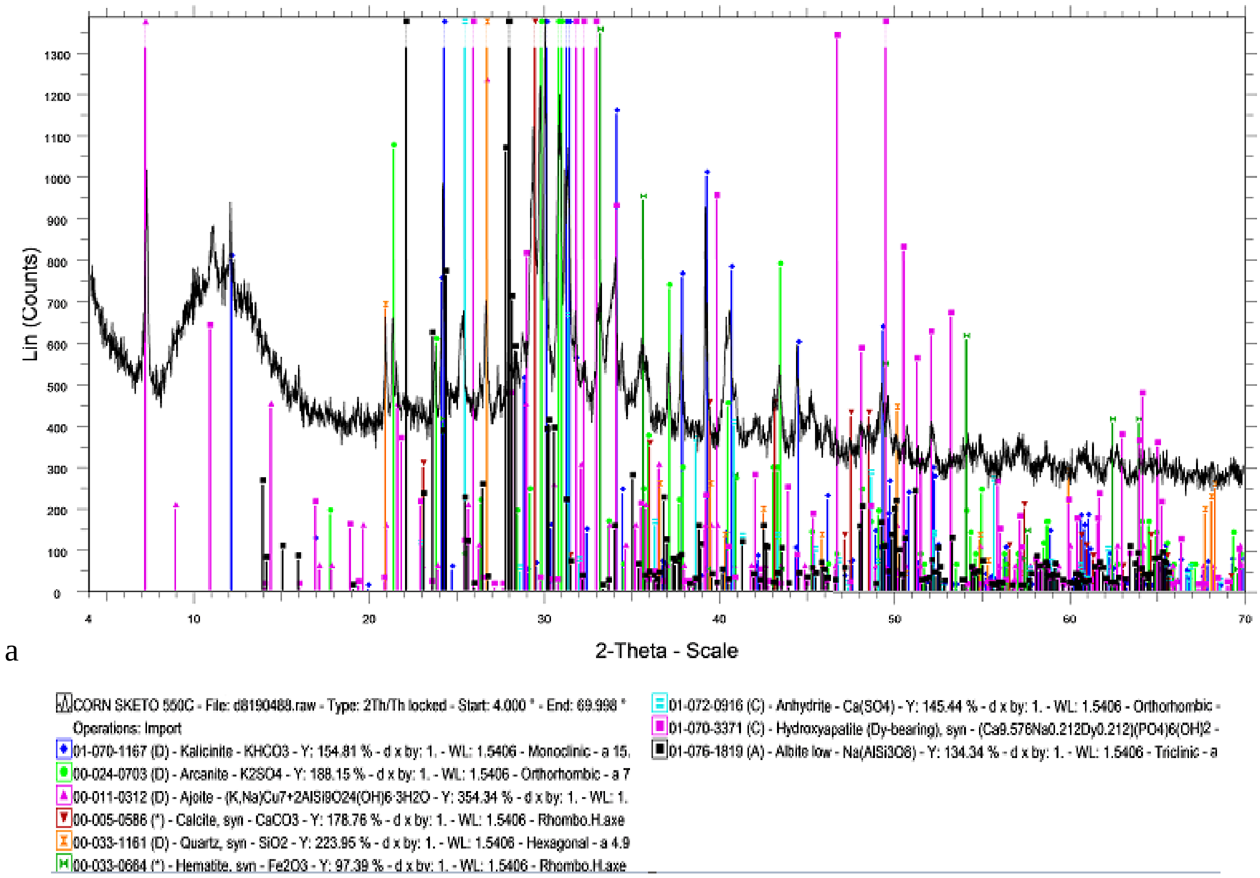
D. Characterization and melting behaviour of ashes of raw fuels and fuel blends

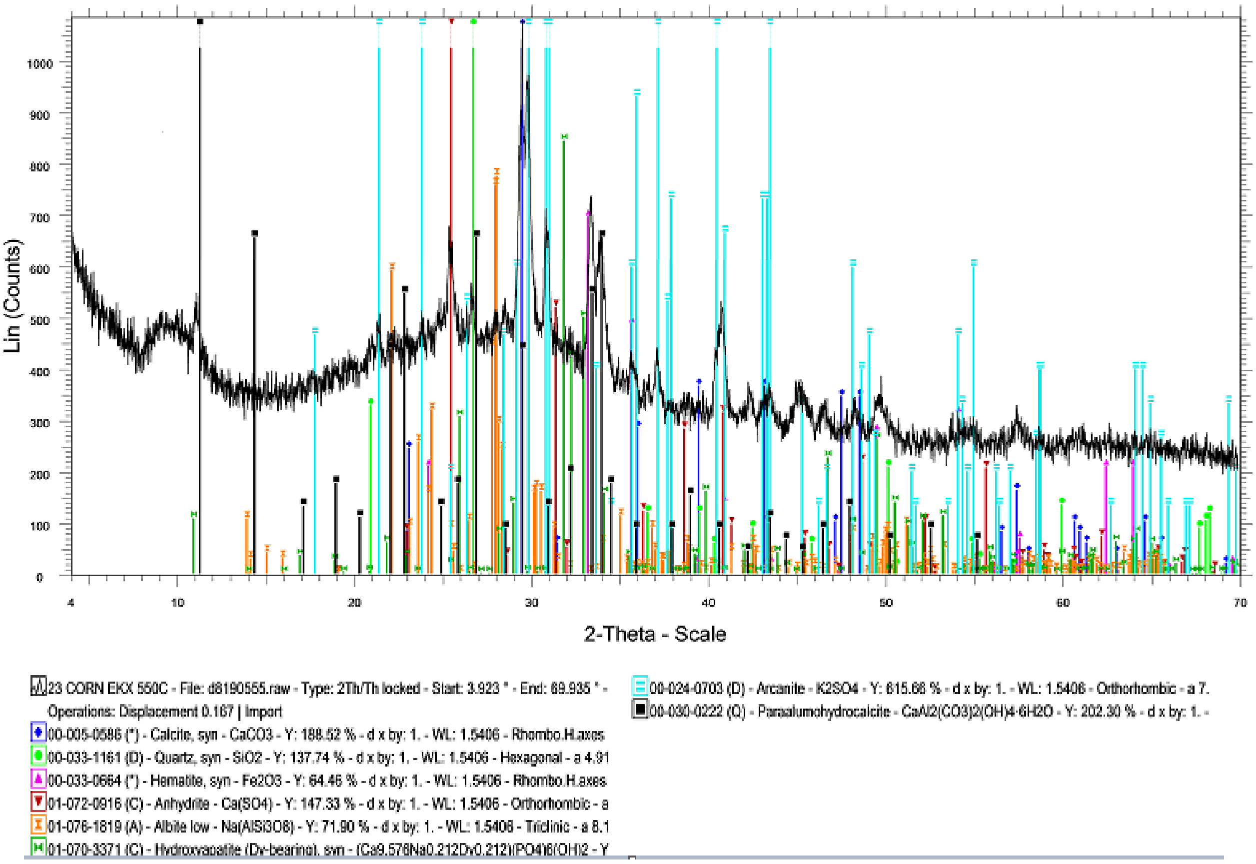
The chemical analysis of ashes of all studied materials is presented in Table IV, typically as oxides of major elements. Biomass samples contained remarkably high concentration of K and significant of P, as opposed to the lignite, most probably due to fertilizers used in agriculture. The percentage of Ca in grape hulls was comparable to that of lignite, whereas the Ca content of corn residues was very low. Lignite ash was richer in Si, Al, Ca, Fe and Mg. When biomasses were washed with water, as can be observed, the concentrations of K and P were reduced by 59-72% and 18-37%, respectively, suggesting that these elements were associated with partially soluble components in water. The composition of ash mixtures varied between that of component fuels.

The XRD spectra (scanning range of 2-70 2θ°, with increments of 0.02°/s) of raw and water leached materials are illustrated in Fig. 5. K-based minerals were dominant in both biomass ashes in the forms of arcanite, kalicinite, fairchildite and sylvite. A significant amount of P was also incorporated in hydroxyapatite, fluorapatite and strouvite. The contributions of quartz and carbonates of Ca and Mg were smaller, while those of aluminosilicates even lower. On the other hand, the ash of lignite which was

TABLE IV. CHEMICAL ANALYSIS OF FUEL ASHES (% WT)

Sample	***SiO_2***	***Al_2O_3***	***Fe_2O_3***	***CaO***	***K_2O***	***Na_2O***	***MgO***	***P_2O_5***
CR	7.0	0.10	0.23	1.6	50.3	1.0	1.2	12.6
CR_l	32.7	0.32	0.90	23.9	20.7	1.2	9.0	10.4
GHC	4.2	3.3	0.79	17.0	34.0	0.39	3.2	9.7
GHC_l	8.1	3.8	1.3	33.9	9.6	0.29	6.0	6.1
LIG	30.3	19.3	5.7	23.4	3.1	1.0	5.5	0.3
LIG/CR	22.0	10.9	3.9	28.5	15.4	0.14	2.7	3.9
LIG/CR_l	30.1	14.4	4.4	33.3	7.5	0.15	4.5	4.2
LIG/GHC	21.3	14.5	5.6	22.3	9.7	0.82	3.8	3.0
LIG/GHC_l	23.9	13.9	3.5	27.0	4.1	0.4	5.5	1.9

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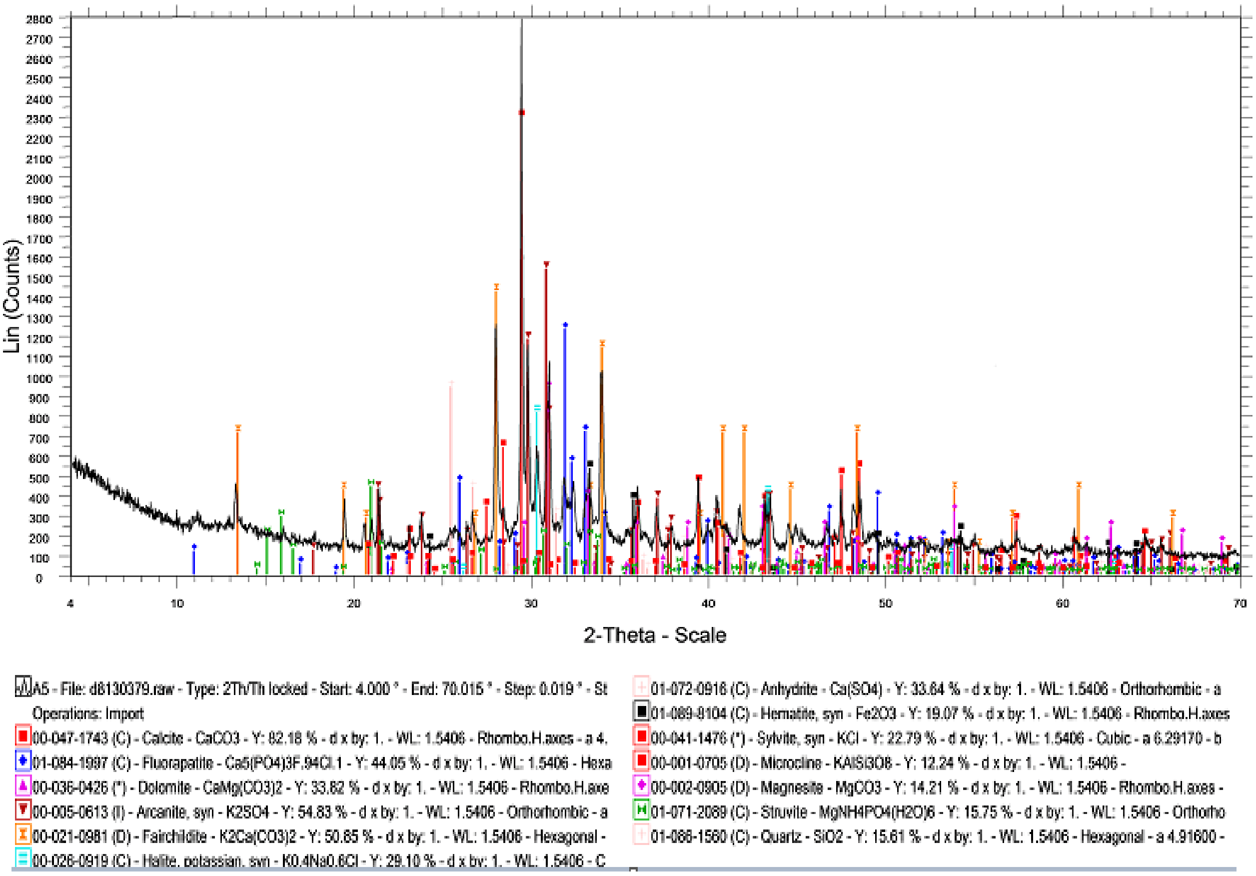
Operations: Displacement 0.167 | Import
00-005-0586 (*) - Calcite, syn - CaCO3 - Y: 188.52 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes
00-033-1161 (D) - Quartz, syn - SiO2 - Y: 137.74 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91
00-033-0664 (*) - Hematite, syn - Fe2O3 - Y: 64.46 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes
01-072-0916 (C) - Anhydrite - Ca(SO4) - Y: 147.33 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a
01-076-1819 (A) - Albite low - Na(AlSi3O8) - Y: 71.90 % - d x by: 1. - WL: 1.5406 - Triclinic - a 8.1
01-070-3371 (C) - Hydroxyapatite (Dy-bearing), syn - (Ca9.576Na0.212Dy0.212)(PO4)6(OH)2 - Y
00-024-0703 (D) - Arcanite - K2SO4 - Y: 615.66 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a 7.
00-030-0222 (Q) - Paraalumohydrocalcite - CaAl2(CO3)2(OH)4·6H2O - Y: 202.30 % - d x by: 1. -

b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00-047-1743 (C) - Calcite - CaCO3 - Y: 82.18 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes - a 4.
01-084-1997 (C) - Fluorapatite - Ca5(PO4)3F.94Cl.1 - Y: 44.05 % - d x by: 1. - WL: 1.5406 - Hexa
00-036-0426 (*) - Dolomite - CaMg(CO3)2 - Y: 33.82 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axe
00-005-0613 (I) - Arcanite, syn - K2SO4 - Y: 54.83 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a
00-021-0981 (D) - Fairchildite - K2Ca(CO3)2 - Y: 50.85 % - d x by: 1. - WL: 1.5406 - Hexagonal -
00-026-0919 (C) - Halite, potassian, syn - K0.4Na0.6Cl - Y: 29.10 % - d x by: 1. - WL: 1.5406 - C
01-072-0916 (C) - Anhydrite - Ca(SO4) - Y: 33.64 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a
01-089-8104 (C) - Hematite, syn - Fe2O3 - Y: 19.07 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes
00-041-1476 (*) - Sylvite, syn - KCl - Y: 22.79 % - d x by: 1. - WL: 1.5406 - Cubic - a 6.29170 - b
00-001-0705 (D) - Microcline - KAlSi3O8 - Y: 12.24 % - d x by: 1. - WL: 1.5406 -
00-002-0905 (D) - Magnesite - MgCO3 - Y: 14.21 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes -
01-071-2089 (C) - Struvite - MgNH4PO4(H2O)6 - Y: 15.75 % - d x by: 1. - WL: 1.5406 - Orthorho
01-086-1560 (C) - Quartz - SiO2 - Y: 15.61 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91600 -

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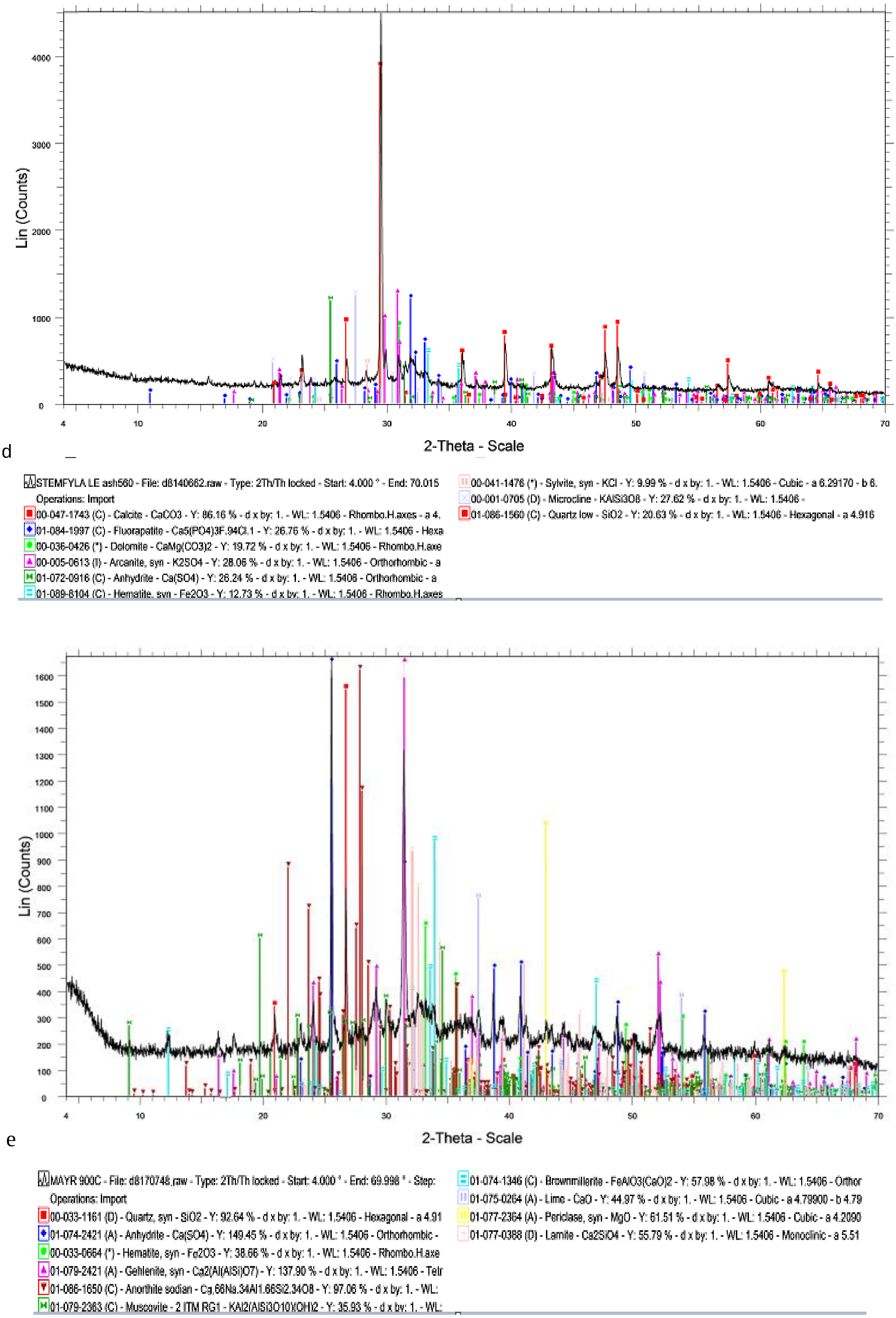

Fig. 5. XRD spectra of (a) CR, (b) CR$_{I}$, (c) GHC, (d) GHC$_{I}$ and (e) LIG

found by XRF analysis to contain higher proportions of Si, Al and Ca, as compared to biomass ashes, presented significant amounts of silicates, aluminosilicates and sulfates in the forms of quartz, gehlenite, anorthite and anhydrite. Fe and Mg occurred in the forms of hematite, brownmillerite and periclase, respectively, whereas larnite, muscovite and lime (generated by decomposition of calcite) were detected in small quantities. Upon leaching of corn residues and grape hulls with water, Fig. 5 shows that some K and P-based minerals, kalicinite, fairchildite and strouvite were dissolved in water, so that they were not identified by XRD, while others, arcanite and hydroxyapatite presented a partial solubility.

The melting behaviour of raw and leached fuel ashes is illustrated in Fig. 6 and the characteristic fusion temperatures, i.e. initial deformation (IDT), softening (ST), hemispherical (HT) and fluid (FT), as recorded by the heating microscope, are compared in Table V. Initial deformation temperatures of raw

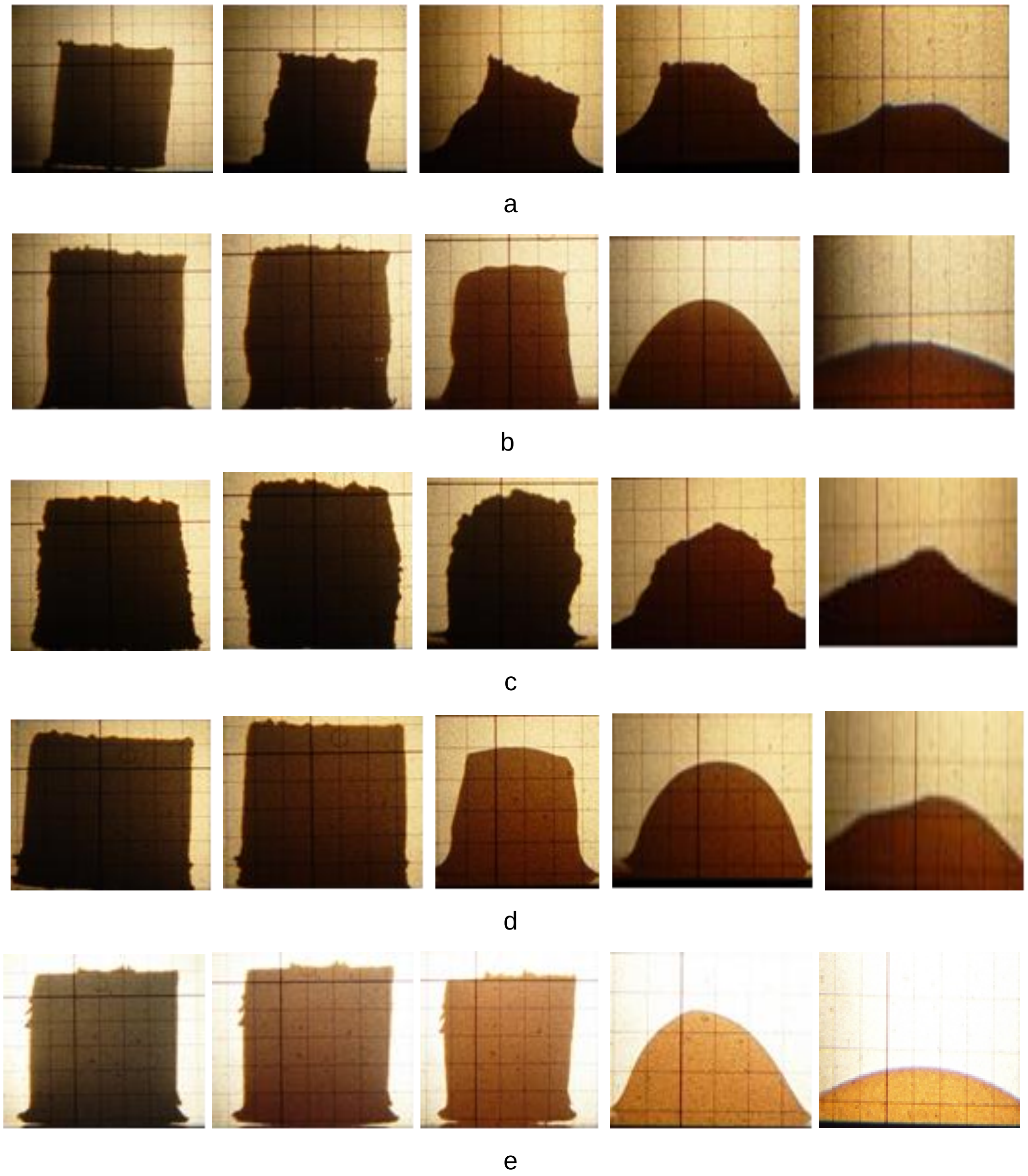

Fig. 6. Melting behavior of ashes of (a) CR, (b) CR$_l$, (c) GHC, (d) GHC$_l$ and (e) LIG

TableV. Fusibility analysis of fuel ashes

Temperature(°C)	***CR***	***CR$_l$***	***GHC***	***GHC$_l$***	***LIG***	***LIG/CR***	***LIG/CR$_l$***	***LIG/GHC***	***LIG/GHC$_l$***
IDT	1006	1080	790	1170	1125	1088	1112	1031	1147
ST	1148	1195	1072	1310	1227	1202	1214	1186	1254
HT	1173	1226	1260	1335	1331	1295	1312	1319	1338
FT	1250	1315	1290	1389	1358	1336	1349	1345	1371

materials, ranging between 790°C and 1125°C, are considered low for most combustion units, apart from fluidized beds operating below 1000°C, revealing deposition problems in boilers. The IDT of grape hulls was particularly low and 335°C lower than that of lignite. The FT of both biomass ashes was also 70°C to 110°C lower with respect to lignite. This behavior of biomass ashes is assigned to their higher concentration in alkali, especially K present in the fusible forms of arcanite (melting point 1070°C), fairchildite (melting point 815°C), sylvite (melting point 790°C), alite (melting point 801°C) and kalicinite (melting point 891°C) [7,22,23], as confirmed by the XRD spectra previously presented. However, the ash of lignite, which contained elevated amounts of Si, Al and Ca based minerals with high melting point, such as quartz (1700°C), gehlenite (1593°C), anorthite (1553°C) and anhydrite (>1300°C) [7,23], whereas a small concentration of alkali, fused at a higher temperature. Furthermore, it is interesting to note that although grape hulls ash had the lower IDT among the samples tested, it had the greatest temperature difference between IDT and FT (500°C), implying a lower fouling rate [7,19]. Table V shows that when alkali minerals of biomasses were dissolved in water after leaching, the IDT of corn residues ash was increased by 74°C and that of grape hulls ash by 380°C, while the FT of corn residues ash was increased by 65°C and that of grape hulls ash by 99°C. Therefore, slagging/fouling problems inside boilers could be reduced.

Concerning the mixtures of lignite with either raw or leached biomass materials, it can be observed that fusion temperatures were intermediate, almost in proportion to each component in the blend and followed the order: LIG/GHC$_l$ > LIG/CR$_l$ > LIG/GHC > LIG/CR. Moreover, the fouling rate of all blends is expected to be similar or even lower in comparison to raw lignite.

IV. CONCLUSIONS

Combustion of corn residues and grape hulls occurred between 200°C and 550°C, whereas that of lignite between 220°C and 600°C. Potassium of biomass materials acted as catalyst during combustion, so that after water leaching the reactivity of the fuels followed the order: CR > CR$_l$ > GHC > GHC$_l$. Mixtures of lignite with corn residues showed a high degree of additivity, while mixtures of lignite with grape hulls exhibited some synergy. Co-combustion of lignite with leached biomass fuels improved its reactivity, which followed the order: LIG/CR$_l$ > LIG/CR > LIG/GHC$_l$ > LIG/GHC. Ash melting of raw materials initiating between 790°C and 1125°C, is considered low for most combustion systems, especially for those fed with grape hulls. When alkali minerals were dissolved in water after biomasses leaching, the initial deformation and fluid temperatures were increased by 74-380°C and 65-100°C, respectively, implying reduced slagging/fouling problems in boilers burning leached fuels or their mixtures with the lignite. Additionally, the fouling rate of all blends is expected to be similar or lower in comparison to lignite.

Acknowledgment *(Heading 5)*

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