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Synthesis and Sintering of High-temperature Composites Based on Mechanically Activated Fly Ash

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Abstract:

Amount of fly ash which is and yet to be generated in the coming years highlights the necessity of developing new methods of the recycling where this waste can be reused in significant quantity. A new possibility for fly ash utilization is in high-temperature application (thermal insulators or/and refractory material products). As such, fly ash has to adequately answer the mechanical and thermal stability criteria. One of the ways of achieving it is by applying mechanical activation procedure on fly ash. In present study, fly ashes from two different power plants were mechanically activated in a planetary ball mill. Mechanically treated fly ashes were cemented with two different binders: standard Portland cement and high-aluminates cement. Physico-chemical analysis and investigation of mineralogical components of composites are emphasized, due to the changes occurred in fly ash during mechanical activation and sintering of composites. Macro-performance of the composites was correlated to the microstructure of fly ash studied by means of XRD and SEM analysis. Thermal stability of crystalline phases was investigated with DTA. Highlight was placed on determination of relationship between mechanically activated fly ash and obtained composites microstructure on one side and behavior of sintered composites on the other side.

Keywords: Fly ash, Mechanical activation, Sintering, High-temperature performance, Recycling.

1. Introduction

The building materials industry is not completely environmentally friendly because it is depleting natural resources. Therefore, the reduction-reuse-recycle principle has to be world-wide adopted [1, 2]. Reduction means minimizing waste through planning and design; Reuse means that the final products can be incorporated back into the same cycle or into another cycle without additional material processing. Recycling refers to the recovery of unavoidable waste, involving chemical or mechanical processing, into secondary materials

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which can be reused. The annual global fly ash production was more than 600 million tons in past 10 years [3]. In the EU, the total production of fly ash is estimated to be about 95 million tons [4, 5]. EU regulations are focused on the recycling of the coal combustion fly ash and reusing it as a component of added-value products [6]. About 21 million tons of fly ash is being reapplied annually in the construction industry [5]. Most of the fly ash is used as concrete/mortar addition [7-10], in road construction [11], as a raw material for cement [12], in bricks and tiles [13-15], for infill of voids and as geopolymers [16-18]. The high recycling rates the fly ash achieved are mainly due to its pozzolanic behavior [19-24].

A new possibility for fly ash application is as raw material for thermal insulators or/and refractory material products. As such, fly ash has to adequately answer on mechanical and thermal stability criteria. One of the ways of achieving it is by applying mechanical activation (MA) procedure on fly ash. Substantial amount of studies related with fly ash performances promotes MA long-term strength enhancement [25-27]. The improved performances are related with the more spherical shape and smooth texture of fly ash compared to cement particles. Fly ash acts as super plasticizing admixture helping the "packing" of composite structure [28]. Super plasticizing ability which leads to reduction of water content needed for optimal mixture consistency and increasing of mechanical strength is only being further enhanced by MA. MA is being applied to minimize the chemical and microstructural incompatibility of fly ashes employed - surface porosity of fly ashes and deviation from spherical particle morphology and to improve reactivity [29, 30]. The importance of employment of fine fly ash on pozzolanic activity and filler effect as the sources of interface reinforcement are reported by various authors [31-37]. Although MA is much more cost effective than application of original sized fly ash, results are by far better. Important aspect of the material is its behavior at elevated temperatures: thermo-insulation properties and refractoriness. The use of fly ash additions in building composites can affect the material behavior when subjected to elevated temperature [38]. Materials that retain a large quantity of water are more desirable for thermal exposure. Some commercial products, used as thermal insulation or passive fire protection in buildings and industrial installations, have a chemical composition and properties similar to fly ash mixtures [39-43].

In the present study, fly ash-cement based composites were subjected to several thermal, mechanical and microstructural analyses in order to investigate behavior under elevated temperature, the mechanical properties and sintering process. Fly ash was previously subjected to MA treatment in order to analyze the impact of grain size distribution and enhanced properties on sintering process of the fly ash based composite.

Experimental procedure

The fly ash used in the investigation originates from the filter systems of two different coal-fired power plants in Serbia. Two representative types of fly ash chosen for the investigation are here labeled as FA-K and FA-TA.

The mechanical activation (MA) procedure was applied on the fly ash samples before mixing fly ash with cement. MA of the fly ash samples was performed in a planetary ball mill device (Retsch-PM4). 2 kg of milling balls were used for milling. Material to milling media ratio of 1:35 was maintained during milling. The fly ash samples were milled for 60 min. Fraction content of the fly ash samples was analyzed by means of cyclo-sizer diffraction particle size analysis (Cyclo-sizer Warman International LTD, Australia). Grain size distribution of the mechanically activated fly ash is given in Fig. 1. Two types of cement were used in the investigation: Portland cement (PC 42.5R Lafarge) and calcium-aluminate cement (CAC Secar 70/71, Lafarge). Chemical composition of CAC and PC is given in Tab. I. Cement-fly ash based composites were prepared containing 30 % of fly ash and 70 % of cement. The composites were labeled as: PCFA-K, CACFA-K, PCFA-TA and CACFA-TA.

Composites were mixed for 8 minutes in laboratory RILEM-CEM mixer and, afterwards, shaped in 2 cm cubic moulds. After 28 days, group of samples was tested on mechanical compressive strength (MCS) at ambient temperature. The rest of samples were sintered in electric furnace at following temperatures: 900, 1100 and 1300 °C, with holding time of 2 hours at each temperature. Heating rate was 150 °C/h. Each group of composite samples was tested for MCS using a conventional laboratory hydraulic pressure device.

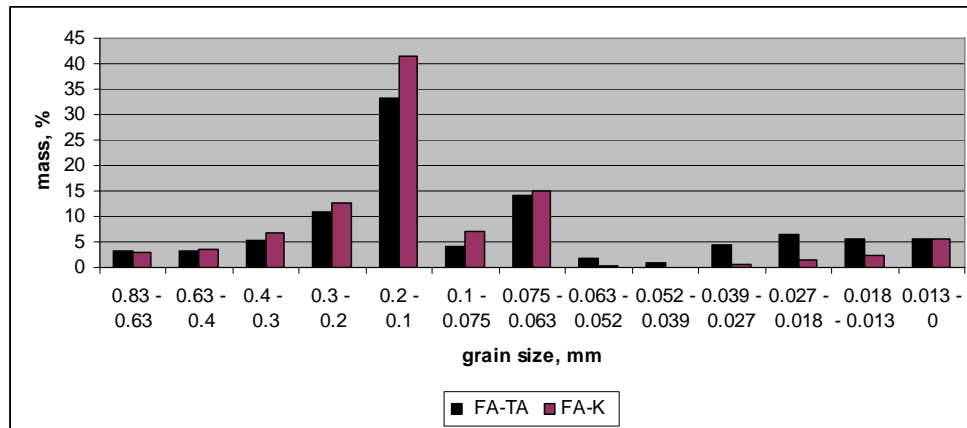


Fig. 1. Distribution of mechanically activated fly ash grain fractions given in percentage of total mass.

X-ray fluorescence (XRF) technique was used to conduct chemical element analysis of the fly ash and composites. Analysis was performed by means of XRF spectrophotometer ED 2000 - Oxford. The chemical composition of fly ash and cements is given in Tab. I.

Tab. I Chemical analysis of fly ash and cement.

Oxides, wt.%	FA-TA	FA-K	PC*	CAC*
SiO ₂	53.45	53.32	19.28	0.11
Al ₂ O ₃	21.28	19.08	6.39	70.85
Fe ₂ O ₃	7.11	6.98	2.93	0.05
TiO ₂	0.56	0.57	<0.10	0.01
CaO	7.61	9.71	59.57	27.73
MgO	2.74	3.30	1.56	0.07
P ₂ O ₅	0.03	0.02	-	-
SO ₃	0.78	1.29	-	-
Na ₂ O	0.44	0.50	0.02	0.28
K ₂ O	1.21	1.16	0.03	0.04
MnO	0.03	0.03	-	-
CO ₂	0.25	0.11	-	-

*(values obtained from manufacturer)

The differential thermal analysis (DTA) of fly ash, cement and composites was performed with a Shimadzu DTA – 50 apparatus. Approximately 30 mg of a sample was used for a DTA testing along with α -Al₂O₃ powder as a reference sample. The sample was heated under an nitrogen atmosphere from 20 up to 1100 °C at heating rate of 10 °C/min. Milled composite samples were analyzed by means of X-ray powder diffraction (XRD). The XRD patterns were obtained on a Philips PW-1710 automated diffractometer using a Cu tube operated at 40 kV and 30 mA. The microstructure of the fly ash samples and composites was characterized by scanning electron microscopy method (SEM) using a JEOL JSM-6390 Lv

microscope. Composites were crushed, and parts of original samples were used in SEM investigation. The samples were covered with gold powder for better reflection to be obtained and measurements performed.

Results and discussion

The chemical composition analysis of the composites is presented in Tab. II. The investigation showed that composites consist mainly of silica, alumina, calcium and iron oxides. Due to the chemical composition of applied cement, composites PCFA-K and PCFA-TA have increased CaO content (approximately 44 %) in comparison with CACFA-K and CACFA-TA whose CaO content is app. 23 %. Al₂O₃ content in CAC based composites is app. 55 %. Application of fly ash increased SiO₂ content in the composites in comparison with starting composition of applied cements. SiO₂ content in all investigated fly ashes was higher than 50 %, while content of Al₂O₃ approximately was about 20 %. Therefore investigated fly ashes can be classified as alumino-silicate ashes, which is the category of ashes attributed with excellent pozzolanic behavior.

Tab. II Chemical analysis of the investigated composites.

Oxides, wt.%	PCFA-K	PCFA-TA	CACFA-K	CACFA-TA
SiO ₂	31.00	29.63	17.50	16.08
Al ₂ O ₃	10.22	10.95	54.59	55.30
Fe ₂ O ₃	4.27	4.10	2.08	1.91
TiO ₂	0.17	0.17	0.16	0.16
CaO	44.64	44.31	23.23	22.93
MgO	2.00	2.13	0.79	0.92
P ₂ O ₅	0.02	0.06	-	-
SO ₃	2.15	2.00	0.32	0.17
Na ₂ O	0.36	0.36	0.35	0.35
K ₂ O	0.76	0.75	0.35	0.35
MnO ₃	0.10	0.08	-	-
CO ₂	0.03	0.02	-	-

Processes taking place during fly ash thermal treatment from 20 up to 1100 °C were identified by means of DTA method (Figs. 2. and 3.). Peak showing at approximately 500 °C is exothermic, corresponding to the transformation of organic matter and the decomposition of CaCO₃. Also, β-quartz undergoes transformation to α-quartz at 573 °C, thus a weak shoulder that follows exothermic peak can be assigned to the presence of quartz in fly ash. The endothermic peak at 900 °C is induced by presence of alumino-silicates. Certain changes in DTA curve above 900 °C pointed to the additional structural changes initiated by sintering process. The peak normally occurring at 1000 °C for fly ash is moved towards 900 °C which is the effect of MA. Namely, temperature of sintering of fly ash is slightly decreased. Melting is not recorded at the temperature 1100 °C which attributes to good refractory characteristics of fly ash as raw component material.

DTA curves of cements are given in Figs. 4. and 5. Detailed interpretation of results obtained for cements is difficult because of complexity of cement composition. The processes of dehydration of individual compounds overlap. Taking into consideration this limitation, following peaks were noted: up to 110 °C - dehydration of AH₃·H₂O gel; peak at 500 °C corresponds to the process of decomposition of aluminate phase - C₁₂A₇ and peak at 950 °C is correlated with decomposition of CA phase. DTA results, as well as XRD results, obtained for PCFA-K and CACFA-K are very similar to the presented ones, so in order to achieve

better visibility of the paper, they are omitted.

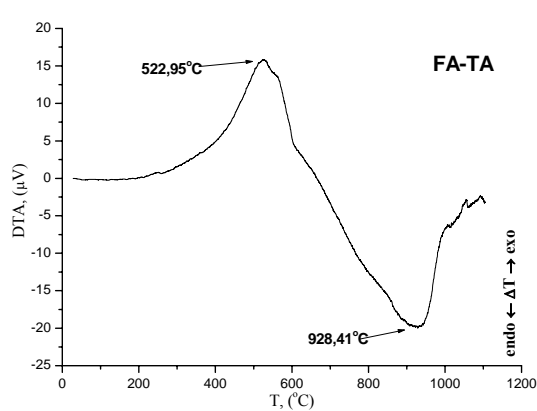


Fig. 2. DTA curve of the FA-TA fly ash.

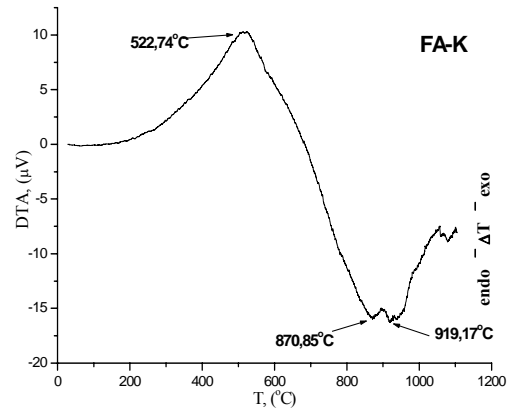


Fig. 3. DTA curve of the FA-K fly ash.

In case of CAC (Fig. 5) the initial amount of water combined with cement is greater and a larger amount of this water is retained at elevated temperatures, particularly up to 300 °C. Dehydration of the calcium aluminates and the alumina hydrates is near completion at 500-600 °C which is marked by endothermic hump on DTA curve. The alumina gel passes through several modifications until it is finally transformed at about 1000 °C into α -alumina. CAH_{10} can be finally dehydrated to CA but other hexagonal hydrates produce calcium oxide and C_{12}A_7 at temperatures from 600-1000 °C. In this temperature range, solid state reactions between calcium aluminates, alumina and lime are induced. This leads to increase in mechanical strength of CAC due to these reactions, which is represented by a small endothermic peak on DTA curve.

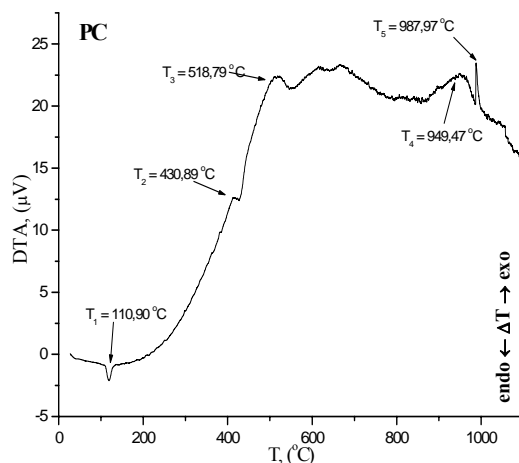


Fig. 4. DTA curve of the PC cement.

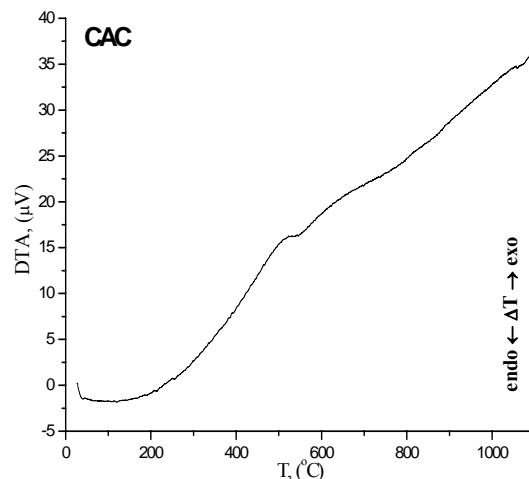


Fig. 5. DTA curve of the CAC cement.

Certain, but not significant quality changes in hydrating systems of investigated cements are being caused by the addition of 30 % of fly ash. Namely, differences in quantity and quality of hydration products cause small differences in recorded DTA curves of cement and cement-fly ash composites (Figs. 6.-9.). In the case of PC the first endothermic effect on DTA is slightly shifted towards 120 °C as a consequence of fly ash addition. At higher temperatures addition of fly ash does not cause significant differences in quality composition of referent cement pastes. In this way a new binders based on waste material with equally good thermal properties are obtained.

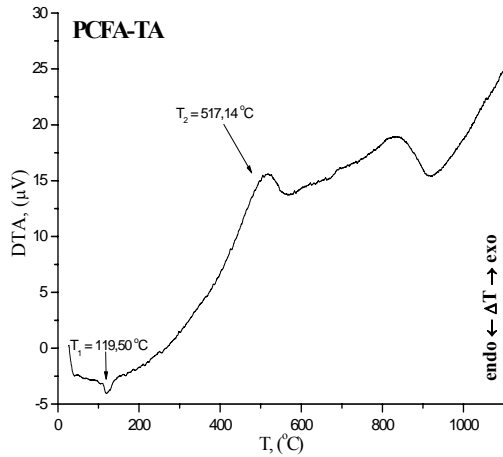


Fig. 6. DTA curve of the PCFA-TA composite.

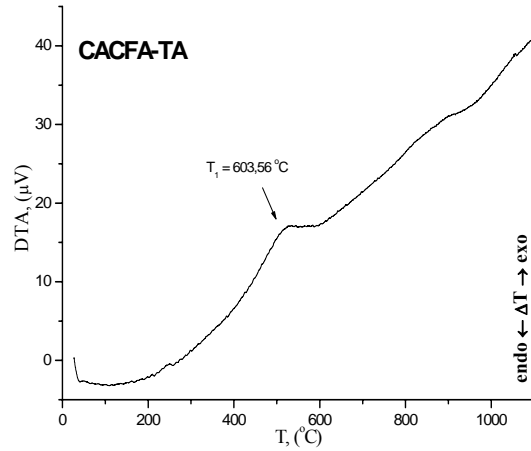


Fig. 7. DTA curve of the CACFA-TA composite.

XRD diffractograms of investigated composites as result of the mineralogical analysis are given in Figs. 8. and 9. Major phases normally present in the PC are: alite, belite, brownmillerite and gypsum. In case of CAC the main present phases are: monocalcium aluminate and monocalcium dialuminate. Fly ash samples contain aluminosilicate glass, quartz and mullite. Magnetite, hematite, fluorite and anhydrite are usually present in relatively negligible amounts.

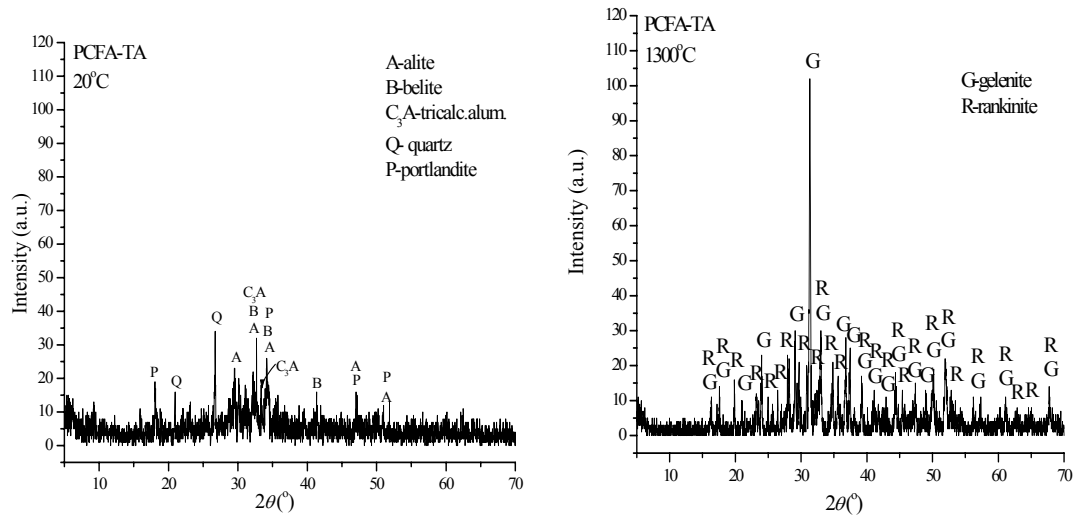


Fig. 8. XRD diffractograms of PCFA-TA at temperatures 20 and 1300 °C.

Phase composition of the composite PCFA-TA (at $T = 20\text{ }^{\circ}\text{C}$) is as follows: alite, belite, tricalcium aluminate (C_3A), quartz (SiO_2), portlandite ($\text{Ca}(\text{OH})_2$). Crystallinity degree of all present phases is very poor. At $T = 1300\text{ }^{\circ}\text{C}$ following phase composition was detected: gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$), rankinite ($\text{Ca}_3\text{Si}_2\text{O}_7$). Besides these crystal phases there is also a small possibility of existence of low amounts of alite and belite. Crystallinity degree is notably higher.

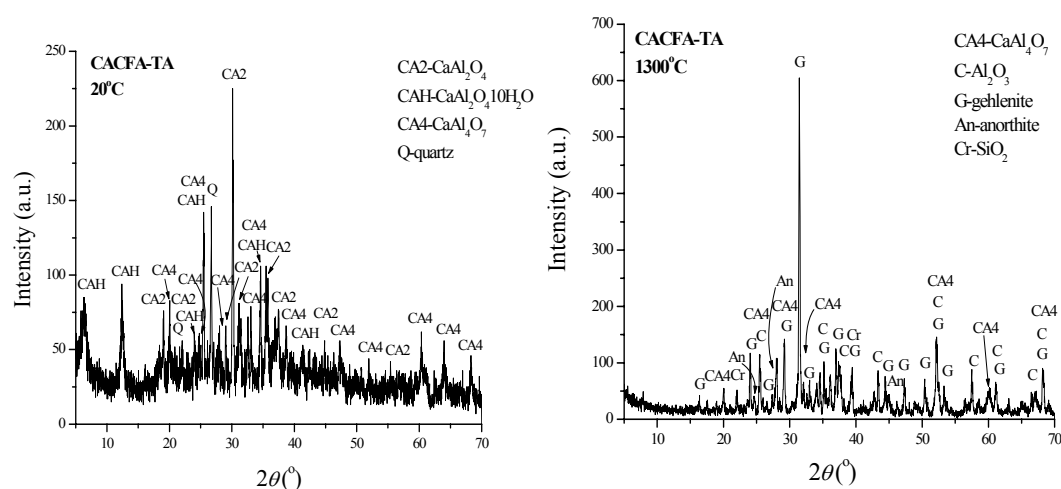


Fig. 9. XRD diffractograms of CACFA-TA at temperatures 20 and 1300 °C.

At $T = 20$ °C, phase composition of CACFA-TA is as follows: monocalcium aluminate, grossite (CaAl_4O_7), calcium aluminium decahydrate ($\text{CaAl}_2\text{O}_4 \cdot 10\text{H}_2\text{O}$), quartz and amorphous matter. The most abundant phase is CaAl_2O_4 , CaAl_4O_7 is less abundant. $\text{CaAl}_2\text{O}_4 \cdot 10\text{H}_2\text{O}$ and quartz are present in small amount. Crystallinity degree of all present phases is very low. At $T = 1300$ °C phase composition is: gehlenite, Al_2O_3 (corundum), CaAl_4O_7 , anorthite, SiO_2 (cristobalite). The most abundant phase is gehlenite, while less present phases are CaAl_4O_7 , corundum and anorthite. There is also possibility of presence of cristobalite but in very small amounts. Crystallinity degree is significantly higher than on the initial sample.

XRD analysis of composites pointed out to certain phase changes occurring with increasing temperature. The XRD spectra in all cases were very complex and the XRD patterns revealed that with an increase of sintering temperature, more complex aluminosilicates were formed, especially in CACFA composites. In case of PCFA composites, the number of the minerals was reduced with an increase of sintering temperature. It is possible that these simple minerals were gradually incorporated into the complex aluminosilicates when the sintering temperature increased. The original ash contained a significant amount of siliceous glass together with the crystalline phases - quartz, hematite and mullite. Sintering reduced the amount of glassy phase and quartz, and promoted formation of anorthite, mullite, hematite and cristobalite. However, the diffraction peaks of these compounds in sintered samples were broad or poorly developed and in some cases overlap. It was observed that, the peak intensities of these compounds slightly increase and their crystallinity improves with increasing temperature. The formation of rankinite, gehlenite, anorthite and cristobalite in the composites is important because they are thermally stable (i. e. have high melting point) and therefore they contribute to the thermal stability of the composites.

The change of compressive strength of the composites after exposure to high temperature is presented in Fig. 10. Obtained values of compressive strengths for all composite types are high, reaching over 100 MPa for CAC and PC composites. Surely, such values should be reduced by dimensional factor, i. e. smaller-sized samples give higher strength values. However, it should be highlighted that mechanical activation of fly ash increased workability of cement paste, decreased porosity and thus improved density of the composites and finally influenced of decreasing of sintering temperature.

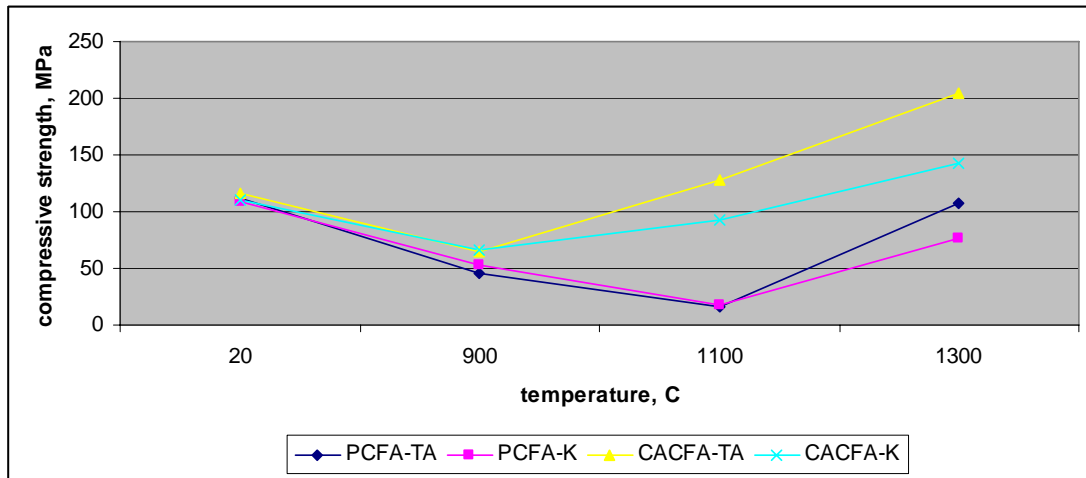


Fig. 10. The change of compressive strength of the composites during exposure to high temperature.

As result, a bonding agent with extremely high strength values and lower sintering temperature is obtained. Both, PC and CAC composites do not show signs of melting process when exposed to high temperatures. Portland cement normally has lower refractoriness than calcium aluminate cement, but the strength values given in Fig. 10, as well as XRD and DTA analyses proved that all investigated composites are highly resistant on high temperature exposure. Also, sintering improved compressive strength of composites. Namely, the strength of cements normally decreases at elevated temperatures due to chemical and physical changes. PC composites showed strength decreasing up to 1100 °C. Afterwards strength started increasing. CAC cements showed characteristic “peak” in strength decreasing around 800-900 °C.

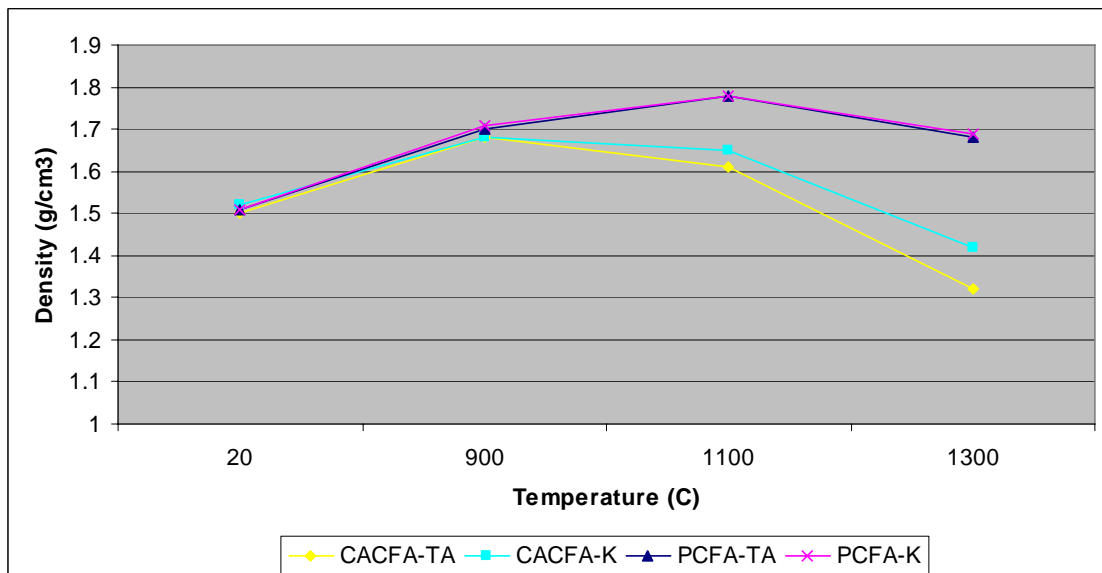


Fig. 11. Density changes during sintering process.

Thus fly ash can be identified as alternative binder which possesses good fire resistance in terms of strength loss at elevated temperature. The first criterion to be considered when selecting a high temperature-resistant aggregate is its thermal stability, both physical and chemical. Thus fly ash can be considered as both bonding agent and small fraction aggregate/filler. That fulfills the purpose of this study, which was to develop a high temperature resistant bonding agent or mortar by using fly ash and proving that it can be applied as high temperature-resistant material.

Fig. 11. shows density changes during sintering at various temperatures for 2 h. Highest values for CAC fly ash - based composites are obtained at 900 °C, and then the slight decrease is noticed at 1100 °C, while the lowest values are after sintering at 1300 °C. On the contrary, densities for PC fly ash - based composites increased during sintering reaching their maximum for 1100 °C and then a slight decreased is observed. These results are in a great accordance with the results that follows.

The SEM micrographs of composites CACFA-TA and PCFA-TA, recorded after samples sintering at $T = 1300$ °C, are given in Figs. 12. and 13, respectively. Porosity of CACFA-TA sample is evidently lower, which explains higher compressive strength of CAC based composites. Porosity and compressive strength of investigated composites were changing with temperatures rising over 900 °C. Decreasing porosity and increasing strength of material usually points out on initiation of sintering process. Mechanical activation destroyed original fly ash particle structure and increased the available surface area. MA contributed in following manner: original fly ash particles gained more spherical and glassy appearance, with particle diameter decreasing and, thus, more easily filling in the voids left behind cement non-spherical particles.

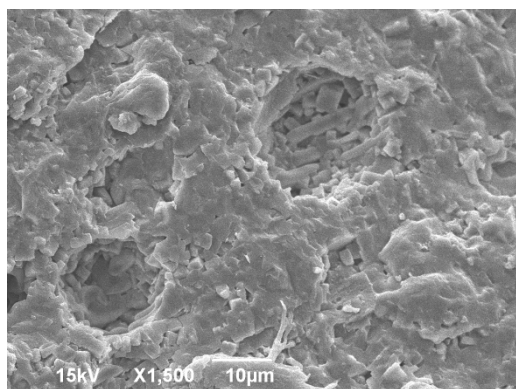


Fig. 12. The SEM microphotograph of CACFA-TA.

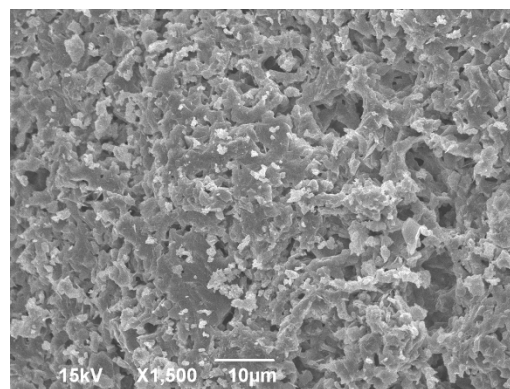


Fig. 13. The SEM microphotograph of PCFA-TA.

It also repositioned crystalline phases within fly ash particles which influenced easier formation of heat-resistant phases in composites. When mechanically activated ash is combined with bonding agent, it improves the composites initial density. Consequently, the compressive strength after sintering is also being improved.

In both presented samples hollow irregularly shaped grains, filled with other smaller spherical particles or microspheres dominate the morphology. Larger particles also may be irregularly shaped. The bond between fly ash and cement particles seems to be quite strong which indicates that these two materials are thermally compatible.

Conclusions

The developed fly ash-cement composites seems to be a promising thermal-insulation and high temperature resistant material which can be used as bonding agent in concretes or mortars for structures and structural elements that are exposed to high temperatures. The investigation highlighted following:

- Mechanical activation promoted long-term strength enhancement and improved over-all performances of investigated composites by minimizing the chemical and microstructural incompatibility of fly ashes and employing it as super plasticizer.
- Fly ash, as raw material, showed positive thermal insulation or temperature protection properties and behavior.
- DTA pointed to the slight shifting of the high temperature peaks (above 900 °C) which means that mechanical activation influenced decreasing of fly ash sintering temperature. Melting of the material is not recorded at the temperature 1300 °C which attributes to good refractory characteristics of investigated composites.
- XRD showed that crystallinity of the composites improved with increasing temperature. The formation of rankinite, gehlenite, anorthite and cristobalite in the composites is important because they are thermally stable and therefore they contribute to the thermal stability of the composites.

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Садржај: Количина летећег пепела који се производи и која ће бити произведена у годинама које долазе захтева развој нових метода рециклирања у којима ће бити употребљене значајне количине овог отпадног материјала. Могућност високо-

температурне апликације (термоизолациони и ватростални материјали) указује на нову могућност за примену летећег пепела. За такву примену летећи пепео мора да задовољи високе механичке и термичке критеријуме. Један од начина да се то постигне је механичка активација летећег пепела. У овом раду приказани су резултати механичке активације пепела из две различите термо-електране помоћу планетарног млина. Активирани пепели су мешани са два везива: портланд цементом и високо-алуминатним цементом. Акцент је на анализи физичко-хемијских својстава и минеролошких фаза због промена које се дешавају у композитима услед механичке активације пепела и синтеровања. Макро својства композита су повезана са микроструктуром испитиваном помоћу СЕМ анализе и Рендгенске дифракције. Термијска стабилност кристалних фаза испитивана је помоћу ДТА. Акцент је на утврђивању корелација између механичке активације летећег пепела и микроструктуре композита на једној и својстава синтерованог композита на другој страни.

Кључне речи: летећи пепео, механичка активација, синтеровање, високотемпературне перформансе, рециклажа.
