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Reactivity of meta-talc in sodium hydroxide medium

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Abstract. This work deals with the reactivity of XRD amorphous delaminated and dehydroxylated talc phase (meta-talc), in alkali medium. Talc ore Luzenac H70 was treated in a stirred ball mill (attritor) for 30; 60 and 90 min with subsequent thermal treatment at 700°C; 750°C and 800°C. Reactivity of mechanochemically and thermally treated talc ore in NaOH solution was studied using isothermal calorimetry. An influence of material crystallinity and amount of water coordinated in material structure after milling on its reactivity in alkali medium was studied.

1. Introduction

Meta-talc (analogically with metakaolin) is an X-ray diffraction (XRD) amorphous delaminated and dehydroxylated talc phase. This new material was prepared at Materials Research Centre at Faculty of Chemistry Brno University of Technology and patented in 2014 [1]. Contrary to metakaolin, meta-talc cannot be prepared by thermal dehydroxylation because of immediate recrystallization of enstatite ($\text{MgO} \cdot \text{SiO}_2$) accompanied with a separation of silica-rich phase at 870–1050°C [1], [2]. Ptáček et al. [1] published the method of preparation of meta-talc by the way of exposition of talc ore to a mechanochemical treatment, i.e. prolonged milling in a high energy milling device (vibration mill, planetary mill, stirred ball mill). Tangential strain induced by the mill caused the delamination and the loss of crystallinity of treated material. Subsequent thermal treatment of delaminated talc phase at temperatures up to 800°C can provide the XRD-amorphous dehydroxylated talc phase, which was termed as meta-talc [1].

With an advantage of real time information, isothermal calorimetry has become a useful technique in studying cement hydration kinetics [3], [4], [5]. Isothermal calorimetry measures the thermal power (heat production rate) produced by the hydration reaction of cementitious and other binding materials. This follows the overall reaction of the binder. The timing and shape of the heat flow curve obtained by the calorimetry is an indicator of relative performance of binding systems. In the isothermal calorimeter, the curing temperature is maintained very close to constant in relatively small sample, which allows a high repeatability and reproducibility [6].

Investigations of alkali activation of metakaolin and slag have displayed that calorimetric measurements are suitable for obtaining the information needed for description of alkali activation processes [7], [8]. Sun and Vollpracht [3] observed broad exothermic peak following initial dissolution peak at calorimetric curves of alkali activated metakaolin and slag. They deduced that after an initial period of dissolution process of alkali activated metakaolin, the concentrations of dissolved ionic species reached a threshold for the outset of following reactions. The dormant period, which corresponded to induction period in cement hydration, was supposed to be a time needed for concentration growing up



[3]. Both polymerization and condensation of geopolymer gels are exothermic processes and made the main contribution to this second peak [3], [9]. In the case of slag the second peak indicated the extensive precipitation of reaction products, such as C-A-S-H [3], [10]. Contrary to that, calorimetric output of NaOH activated fly ash showed only single extensive peak. The slow cumulative heat increase could be assigned to the continuous dissolution and slight transformation of dissolved species into gels [3].

This paper is focused on observation of alkali activation of meta-talc using isothermal calorimetry. Effect of mechanochemical activation period and thermal treatment temperature on the reactivity of meta-talc samples was assessed in order to optimize the preparation of this new material for future experiments.

2. Experimental

For the purposes of this work talc ore Luzenac H70 from Imerys was used. Chemical composition and loss of ignition of starting material are reported in Table 1.

Table 1. Chemical composition and loss of ignition of talc ore Luzenac H70.

substance	content (wt. %)
SiO ₂	45.29
MgO	31.72
Al ₂ O ₃	11.86
Fe ₂ O ₃	1.27
CaO	0.52
LOI	9.06

Phase composition, obtained by X-ray diffractometry with use of Rietveld refinement, is shown in Table 2. Unfortunately, due to a preferred orientation of crystals in examined material it is not possible to determine the correct phase composition. Nevertheless it is possible to state that the chosen talc ore consisted mainly of clinocllore – chlorite type phyllosilicate with high Mg content. The second significant phase is talc. The rest phases are present in content of less than 10%.

Table 2. Phase composition of talc ore Luzenac H70.

phase	content (wt. %)
clinocllore	59.4
talc	29.8
quartz	9.0
muscovite	1.5
dolomite	0.3

2.1. Preparation of samples

A batch of 100 g of talc ore was milled in a stirred ball mill Netsch Attritor for 30; 60 and 90 min. Milling balls with diameter of 7 mm were chosen as milling media. The weight of milling balls was 7 300 g. The stirrer rotation speed was 500 rpm. Mechanochemically treated samples were calcinated at 700°C; 750°C and 800°C. The sample was heated with a rate of 5 °C·min⁻¹ up to the required temperature. After being held for 1 hour at temperature of calcination, the crucible with sample was

cooled with a cooling rate of $5^{\circ}\text{C}\cdot\text{min}^{-1}$ to 200°C and finally cooled to room temperature in a desiccator. Phase composition of all samples was characterized by X-ray diffractometer Empyrean (PANalytical).

2.2. Isothermal calorimetry

Samples of mechanochemically activated talc ore without thermal treatment and samples after calcination at above mentioned temperatures were treated in alkali medium. A weight of 2 g of sample was mixed with a volume of 1 cm^3 of $19\text{ mol}\cdot\text{dm}^{-3}$ NaOH solution. Time dependence of heat flow and evolved heat was measured for 60 hours in isothermal calorimeter TAM Air (TA Instruments).

3. Results and Discussion

3.1. XRD characterization of prepared samples

As depicted in Figure 1, mechanochemical activation of talc ore led to significant reduction of crystallinity of studied material. Structures of main phyllosilicate phases, clinochlore and talc, were disrupted due to strain whilst quartz phase was not affected during milling due to tougher covalent structure. Calcination at 700°C caused a shift of peak intensity from hydrated clinochlore to a dehydrated one (decrease of intensity of clinochlore peaks at 12.46° , 18.74° and 25.08°). After thermal treatment at 750°C the clinochlore phase was mostly decomposed; peak of dehydrated clinochlore at 6.23° disappeared. At 800°C partial crystallization of high temperature phases, such as enstatite, forsterite or diopside, took place. We can see that sample milled for 30 min had lower content of high temperature crystalline phases after thermal treatment in comparison with longer milled samples. It was caused by larger particle size of this sample which resulted in less proper thermal treatment.

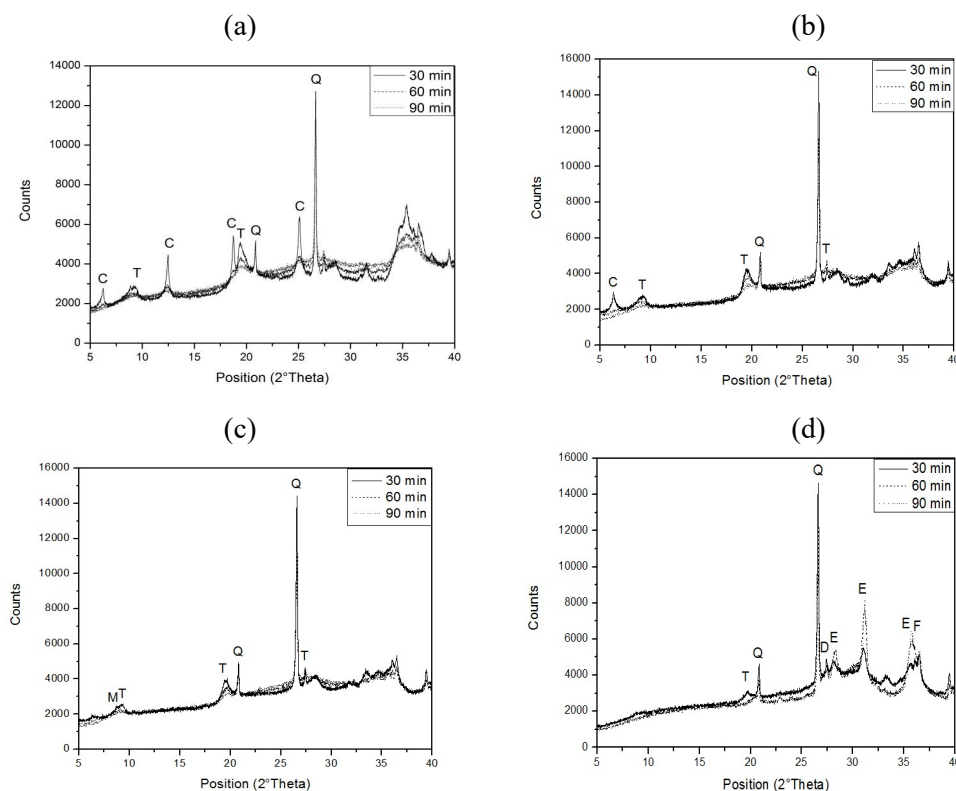


Figure 1. Influence of mechanochemical activation and thermal treatment a) none; b) at 700°C ; c) at 750°C ; d) at 800°C on phase composition of talc ore (C – clinochlore, T – talc, Q – quartz, M – muscovite, E – enstatite, F – forsterite, D – diopside).

3.2. Isothermal calorimetry study of meta-talc reactivity in NaOH solution

Based on the Figure 2, one can consider that reaction between mechanochemically activated talc and NaOH solution takes places quite intensively and about 10 hours after mixing the reaction rate lowers significantly. The reactivity of mechanochemically activated product increases with prolonged milling period due to gradual crystallinity and particle size reduction despite the fact that intensity of heat evolution in first minutes contrarily grows with shorter milling times.

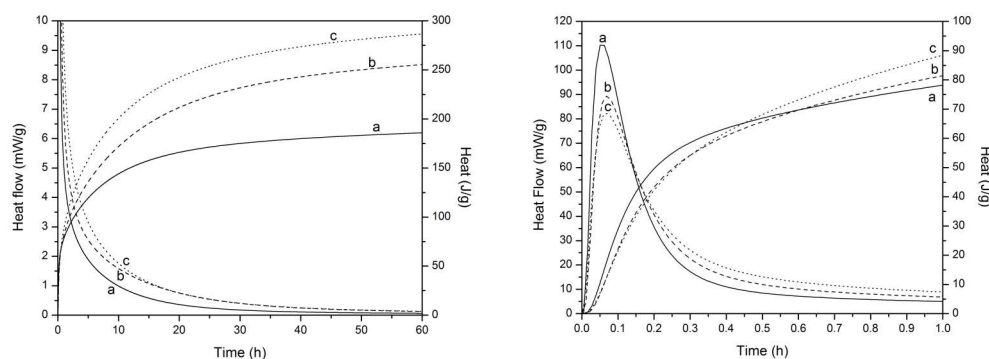


Figure 2. Heat evolution of talc ore mechanochemically activated for a) 30 min; b) 60 min; c) 90 min.

Thermal treatment at 700°C led to noticeable changeover in reactivity of delaminated talc ore (Figure 3). Initial dissolution period and generally heat evolution in first hours were less intensive and whole reaction course was considerably more gradual. But on the other hand amount of evolved heat after 60 hours of reaction was comparable with the case of uncalcinated talc ore. We can consider that the transformation of meta-talc treated at 700°C into reaction products was relatively slow. These results are similar to the previous one of alkali activated fly ash [3].

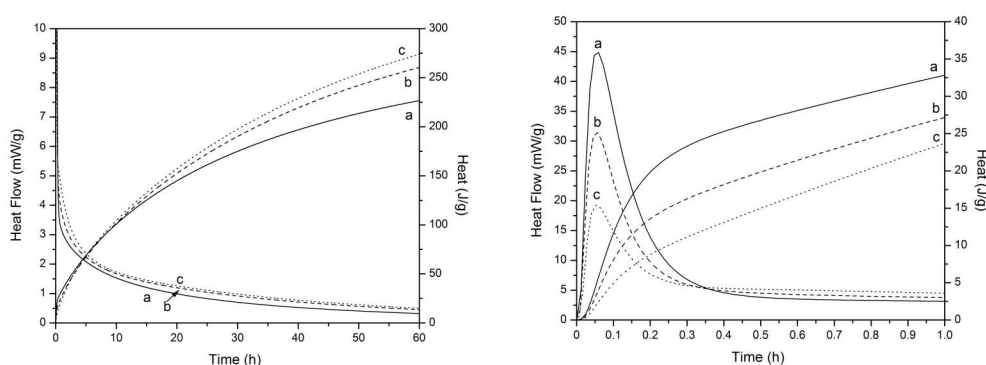


Figure 3. Heat evolution of talc ore mechanochemically activated for a) 30 min; b) 60 min; c) 90 min after thermal treatment at 700°C.

Results depicted in Figure 4 indicate that calcination at 750°C brings about further slowing down of alkali activation of studied samples. We can also observe a quite broad additional peak following immediately after initial period. In this case the course of heat evolution curves is more similar to the curves of alkali activation of metakaolin [3]. Overall amount of evolved heat after 60 hours is comparable with previous two cases except of talc ore milled for 30 min. Reactivity of this sample was closer to reactivity of longer milled samples in opposite to the case of samples calcinated at 700°C. It

was caused by more intensive removing of water molecules coordinated in material structure after mechanochemical treatment.

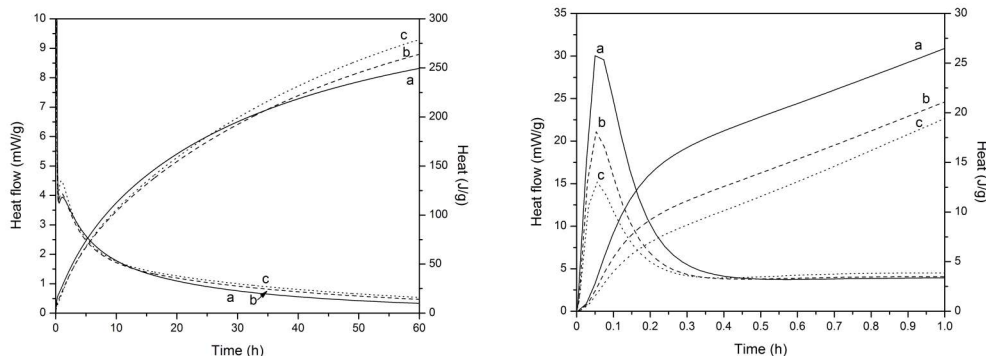


Figure 4. Heat evolution of talc ore mechanochemically activated for a) 30 min; b) 60 min; c) 90 min after thermal treatment at 750°C.

As depicted in Figure 5, thermal treatment at 800°C resulted in strong deterioration of material reactivity due to crystallization of stable high temperature phases. In contrary to other three cases sample milled for 30 min was decidedly most reactive due to less proper calcination which corresponds with X-ray diffraction outputs shown in Figure 1.

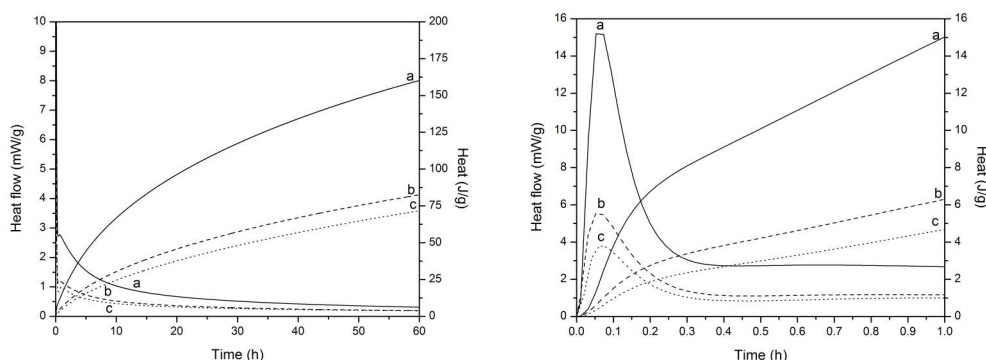


Figure 5. Heat evolution of talc ore mechanochemically activated for a) 30 min; b) 60 min; c) 90 min after thermal treatment at 800°C.

4. Conclusion

Talc ore Luzenac H70, consisting predominantly of clinocllore and talc, was mechanochemically activated in a stirred ball mill for 30; 60 and 90 minutes and subsequently calcinated at 700°C; 750°C a 800°C.

It was found out that calcination at 700°C and 750°C prolongs reaction period where gradual course of reaction is advantageous for preparation of alkali activated materials. Most significant difference between these two temperatures of thermal treatment were observed in the case of a curve of material activated for 30 minutes where thermal treatment at 750°C lowered the reactivity deficit against samples milled for longer periods. However, calcination at 800°C led to formation of stable crystalline phases (enstatite, forsterite and diopside) and related substantial deterioration of reactivity of all three samples. Rate of reactivity deterioration was dependent on milling period and related particle size of studied material.

Acknowledgement

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