Preparation of meta phase of kaolinite as a precursor for geopolymer adsorbent fabrication

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Abstract
Calcined kaolinite was produced by thermal treatment of raw kaolinite obtained from high quality kaolin deposits intercalated sedimentary rocks in south Sinai (Egypt). The optimal parameters for complete de-hydroxylation of kaolinite were achieved. It was found that heating at 700 °C for 90 minutes was suitable for activation of kaolinite. The formation of meta-kaolinite was investigated by XRD, IR analyses of raw kaolinite and meta-kaolinite. The pozzolanic activity was determined by Chappell test, the obtained average value of 0.70 g $Ca(OH)_2$/g meta-kaolinite indicated that the produced meta-kaolinite can be used as an active source for fabrication of geopolymer adsorbent material.
Keywords: kaolinite, meta-kaolin, pozzolanic activity
Kulcsszavak: kaolinit, meta-kaolin, puccolán aktivitás

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1. Introduction

Egypt has several varieties of kaolin deposits at South Sinai and Aswan (Upper Egypt), these locations are good sources for production of meta-kaolinite. The main process for the production of meta-kaolinite as high reactive pozzolan from clay minerals is calcination. The heating process removes water from kaolin, resulting in an amorphous aluminum silicate. This process is known as de-hydroxylation [1] as exemplified in (*Eq. 1*).

$$Al_2O_3{\times}2SiO_2{\times}2H_2O \rightarrow Al_2O_3{\times}2SiO_2 + 2H_2O \quad (1)$$

Thermal treatment of kaolin has been subject of some investigations [2-5]. Heating parameters such as temperature, heating rate and time significantly affect meta-kaolinite formation. For evaluation of the performance of meta-kaolinization by thermal treatment the degree of de-hydroxylation (D_{tg}) [5] is estimated according to equation (*Eq. 2)*.

$$D = 1 - (M/M_{max}) \quad (2)$$

Where, M is the difference between the loss of ignition at 950 ±50 °C (M_{max}) and the loss at certain temperature. The de-hydroxylation of pure kaolin ($Al_2O_3{\times}2SiO_2{\times}2H_2O$) at ambient atmosphere results in mass loss of about 14% and D = 1, which corresponds to the mass of bound hydroxyl groups in kaolin.

The heating temperature for production of the reactive meta-kaolinite phase is usually in the range of 600-800 °C and this temperature plays important role in the reactivity of meta-kaolinite produced. Meanwhile, more heating, causes re-crystallization resulting in mullite mineral formation ($3Al_2O_3{\times}2SiO_2$) causing loss of reactivity [6-8].

The reactivity of meta-kaolinite is measured by its reactivity to react with $Ca(OH)_2$ in the presence of water to form compounds that have cementitious properties. This reactivity can be determined chemically by measuring of the amount of lime consumed, which is determined by various techniques such as thermographic analysis (TGA), differential thermal analysis (DTA), X-ray diffraction (XRD) and calorimetric analysis [9-11]. Also, it can be determined indirectly by measuring the compressive strength developed by the reaction of MK with alkaline silicate reactive solution.

Geo-polymer is a type of amorphous alumino-silicate cementitious material which can be produced by polymerization reaction of aluminum silicate materials with alkaline solutions. Geo-polymeric concrete can be produced by using of pozzolans, which are materials obtained from various activities as byproducts, such as type-F fly ash, low calcium fly ash and calcined Sidorajo volcanic mud [12].

Several varieties of geopolymer have been exploited from modern inorganic chemistry, physical chemistry, mineralogy, geology, analytical chemistry and engineering process technologies. The potential applications of geopolymer include refractory, low energy ceramic tiles, plates for aircraft interior and automobile decoration artifacts, thermal insulation, thermal shock refractory, cements and concretes, composites for infrastructures repair and strengthening, high-tech. resin systems, radioactive and toxic waste containments, cultural heritage and archaeology [13].

The present study aims to determine the optimum conditions for thermal activation of kaolinite for the production of meta-kaolinite using local kaolin deposits in south Sinai, Abu Zenima area for using it for the preparation of geopolymer adsorbent.

2. Experimental methods and materials

2.1 Materials

Representative samples of kaolin were collected from Abu Zenima, South Sinai. Samples were dried to remove humidity, crushed to a particle size of 3 mm. The chemical and physical characteristics were determined in the Institute of Raw Materials and Building Technology that follows the National Center for Building and Housing, using X-ray Fluorescence (XRF) apparatus (XRF Bruker S4 Pioneer) it was operated at 60 kv, and the X-Ray Diffraction (XRD) analysis using an X-Ray Diffractometer (Model Bruker D8 Advance) to determine the mineralogical structure. The metakaolin was grinded to powder of mean particle size of 4.5 μm (*Fig. 2*), to be more active in polymerization reactions. Particle size distribution was performed using particle size distribution analyzer. Differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) were performed using Lenseis Thermobalance instrument.

2.2 De-hydroxylation procedure

Samples of 50 g were heated in a laboratory furnace to different temperatures (100, 200, 300, 350, 400, 450, 500, 550, 600, 650, 700, 750, 800 °C) at different heating durations. The samples were cooled to room temperature to avoid recrystallization. The difference in weights before and after heating was determined in order to calculate the weight loss after thermal treatment.

The activity of meta phase of thermally treated samples was evaluated according to Chappell test [14]. 1 g of meta-kaoliniteite was mixed with 1 g of lime ($Ca(OH)_2$ in 200 ml boiling water. The suspension was subsequently boiled for 16 h and free $Ca(OH)_2$ was determined using sucrose extraction and titration with HCl.

3. Results and discussion

In the present work, it was found that the high quality raw kaolin containing about 80% kaolinite and loss of ignition (L.O.I) at temperature of 950 ± 50 °C is about 12.5% leaving 32.2% Al_2O_3 and 51.2% SiO_2. The chemical compositions of K and MK were detected by using XRF analysis and they are listed in *Table 1*. The mineralogical characteristics of the prepared MK are shown in *Fig. 1a*. XRD analysis showed that there is no mineral in the prepared MK samples, just only quartz impurities in the raw kaolin.

XRD pattern showed the disappearance of kaolin peaks as compared to the raw kaolin (*Fig. 1b*). The major minerals of kaolin deposit are kaolinite and quartz. XRD of heated kaolin at different temperatures are given in *Fig. 1a*, after thermal treatment of kaolin at 550, 600, 650, 700 and 750 °C and heating period. The characteristic peaks of kaolinite (2θ 12.41, 20.21 and 25.49°) disappeared, while the peaks assigned for Qz (2θ 21.22 and 27.45°) remained unchanged.

Particle size-distribution of the grinded MK is plotted in *Fig. 2*, which showed that the diameter of MK in cumulative % is 90% of ~8.560 μm and 10% of ~1.262 μm with an average diameter of ~4.507 μm.

Component	Content, mass (%)	
	K	MK
SiO_2	52.0	55.3
Al_2O_3	32.3	36.0
Fe_2O_3	1.12	1.41
K_2O	0.03	0.07
Na_2O	0.12	0.16
CaO	0.29	0.51
MgO	0.16	0.19
TiO_2	2.13	3.24
P_2O_5	0.16	0.12
Cl	0.08	0.11
SO_3	0.33	0.24
L.O.I.	11.5	0.2
Total	99.70	99.53
Density (g/cm³)	2.60	2.65

Table 1 Chemical and physical characteristics of K and prepared MK
1. táblázat K és MK kémiai és fizikai tulajdonságai

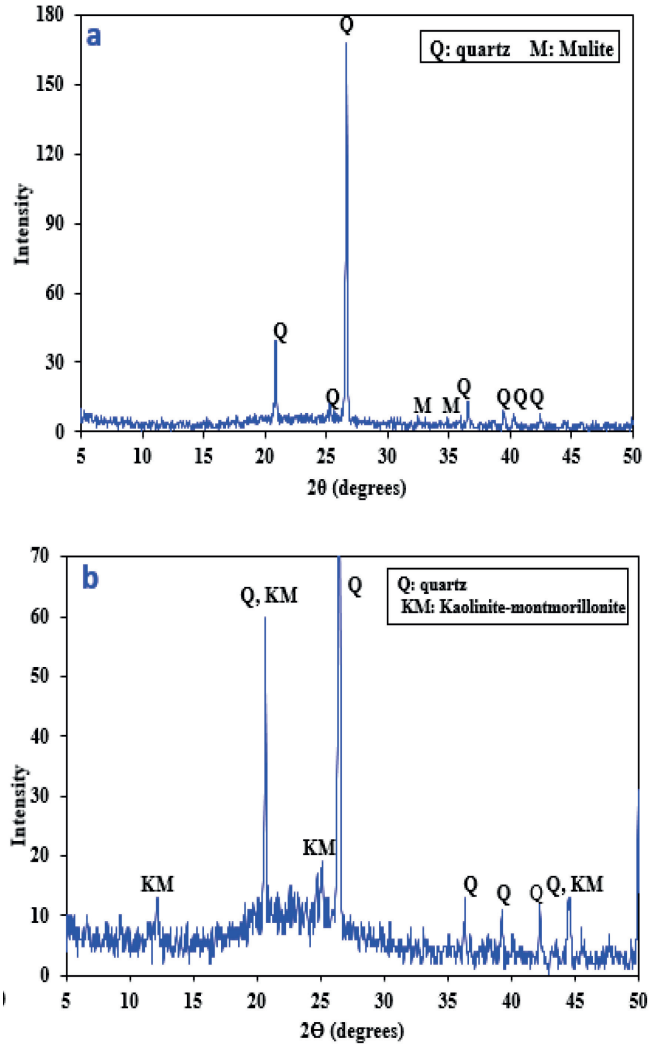

Fig. 1 XRD of (a) metakaolin treated at 700 °C and (b) kaolinite
1. ábra 700 °C-ra hevített (a) metakaolin és (b) kaolinit XRD vizsgálatának eredményei

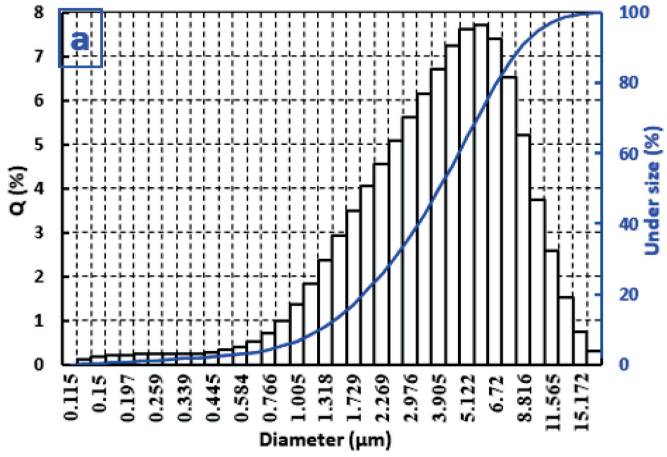

Fig. 2 Particle size-distribution of the grinded MK
2. ábra Az őrölt metakaolin szemcseméret eloszlása

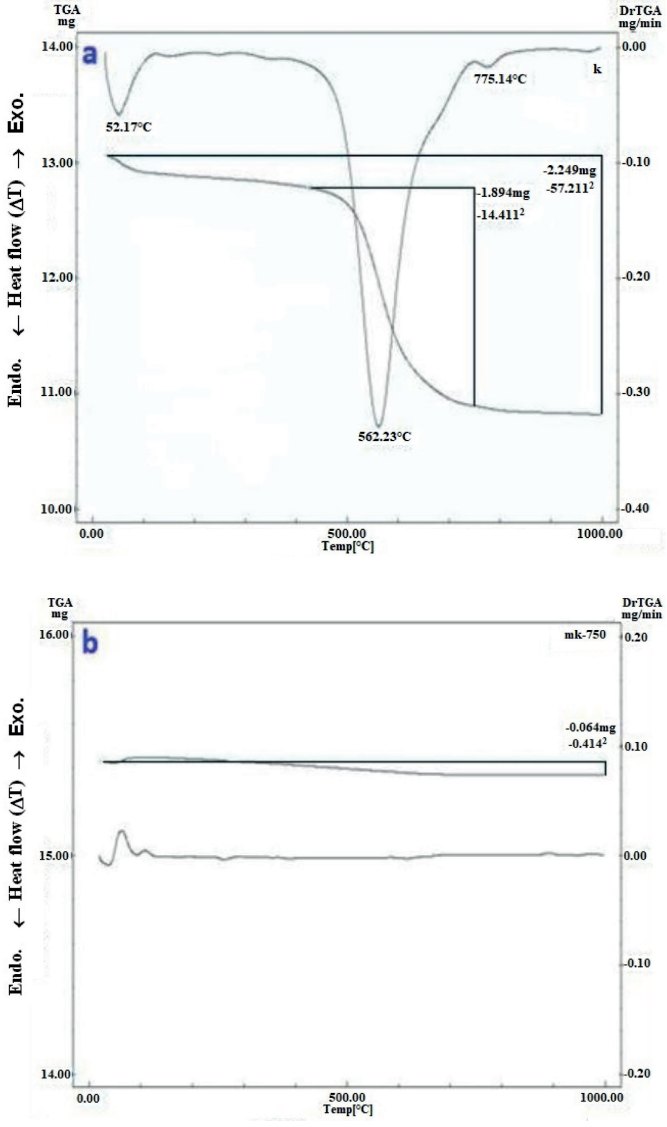

Fig. 3 Thermal gravimetric analysis (TGA/ DTGA) of (a) raw kaolinite and (b) metakaolinite after heat treatment at 750 °C for two hrs
3. ábra (a) nyers kaolinit és (b) metakaolinit termogravimetriás analízisének eredményei (TGA/DTGA) 750 °C-on történő két órás hőkezelés után

Metakaolinite was used as starting material for laboratory synthesis of geopolymer and was generated by thermal activation of kaolinite clay. *Fig. 3a* shows that the thermal activation of clay minerals is in the temperature range between 100 and 800 °C results generally in the de-hydroxylation of the clay mineral. At temperature below 200 °C, the absorbed water in the pores and on the surface was released. Between 200-450 °C, the mass loss is attributed to the pre-hydroxylation process, as a result of re-organization of the octahedral layer. In the temperature range 450-650 °C, de-hydroxylation of kaolinite and formation of meta-kaolinite occurs, while at about 1000 °C, mullite was formed, as indicated by exothermic peak. The observed endothermic peak with a maximum at 550 °C is attributed to the de-hydroxylation process [15].

The octahedral sheet loses water and decomposes into a disordered meta-state in case of collapsing clay minerals [15].

The meta-stable state of the collapsing clay is generally known to be reactive called pozzolana [16, 17]. *Fig. 3b* shows firing the clay to higher temperatures resulting in the formation of new phases such as spinel and mullite [18, 19]. Most studies have proven the utilization of secondary (waste) resources in the synthesis of geopolymer cements or concretes, such as fly ash or lava from coal and slag [20, 21].

Noteworthy, the fundamental unit of the kaolinite structure is composed of an aluminum octahedral sheet and a silicon tetrahedral sheet. Interlayer hydroxyl groups extend from the octahedral sheet into the interlayer region where they form hydrogen bonds to basal oxygens of the opposing tetrahedral silicate sheet [22, 23] (*Fig. 4*).

The first attempt to collect a crystallographic model of metakaolinite was performed by Brindley and Nakahira [24, 25] (*Fig. 5*). It was reported that, during the de-hydroxylation process the octahedral layer is likely to be changed more than the tetrahedral silica layer. Proposed structure of the metakaolinite displays no OH groups.

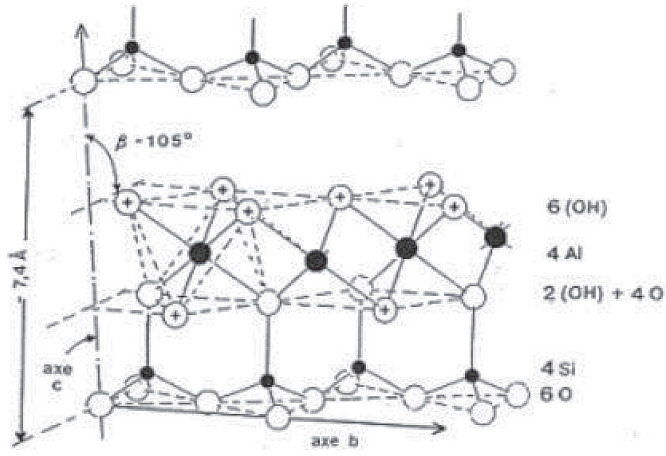

Fig. 4 Chrystalochemical structure of kaolinite [22]
4. ábra A kaolinit krisztokémiai szerkezete [22]

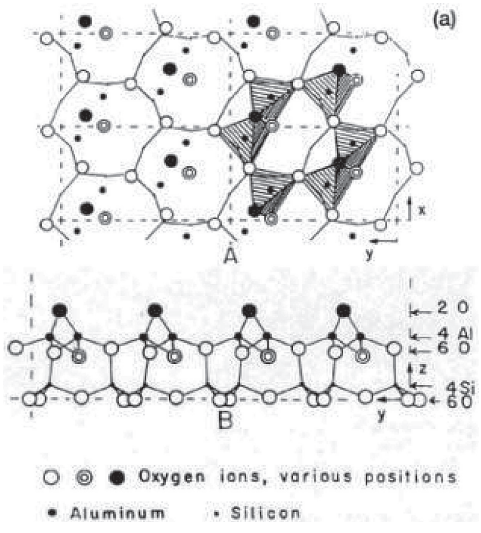

Fig. 5 Lattice of metakaolinite supposed by Brindley and Nakahira [24, 25]
5. ábra Brindley és Nakahira által javasolt metakaolinit rács [24, 25]

The aforementioned knowledge encourages us to believe that the formation of metakaolinite was produced through a stepwise mechanism as depicted in *Fig. 6*.

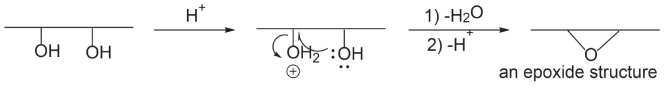

Fig. 6 A plausible mechanistic for the formation of metakaolinite
6. ábra A metakaolinit képződésének feltételezhető mechanizmusa

IR spectroscopy was performed to confirm kaolinite transformation during thermal treatment. IR spectra of raw and thermally treated kaolinite are shown in *Fig. 7a-7d*. The characteristic bands of kaolinite were presented and interpreted as reported in references [26, 27] as follows: OH^- at 3700, 3620 cm^{-1}, Al-OH at 913 cm^{-1}, Si-O at 1032, 1008, 470 cm^{-1} and Si-O-Al at 538 cm^{-1}. Al-OH band at 913 cm^{-1}, and doublet at 3700 cm^{-1} and 3620 cm^{-1}, were disappeared, in *Fig. 7b-7d*, the bands at 539 and 913 cm^{-1} and appearance of new band at 800 cm^{-1} refers to the conversion from octahedral coordination of Al(III) in kaolinite to tetrahedral coordination in meta-kaolin. The bands at 1100 and 1200 cm^{-1} are assigned to amorphous silicate.

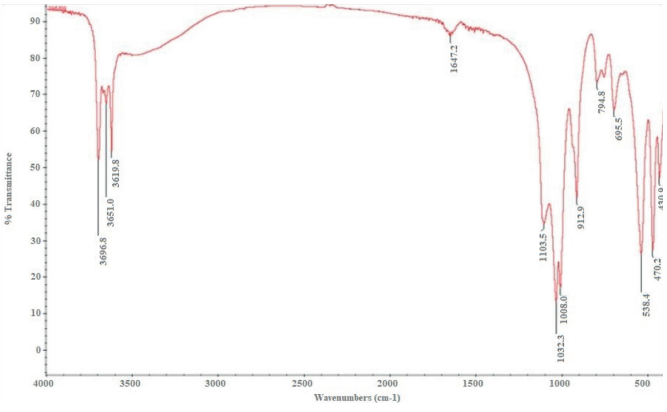

Fig. 7a IR spectrum of kaolinite
7a. ábra A kaolinit IR-spektruma

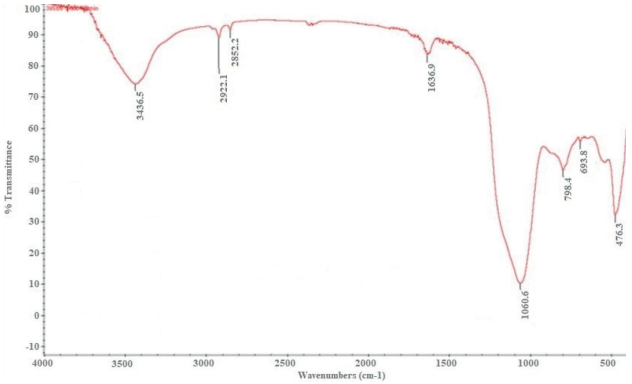

Fig. 7b IR spectrum of thermally treated kaolinite at 550 °C for 90 min
7b. ábra Az 550 °C-on 90 percig hőkezelt kaolinit IR spektruma

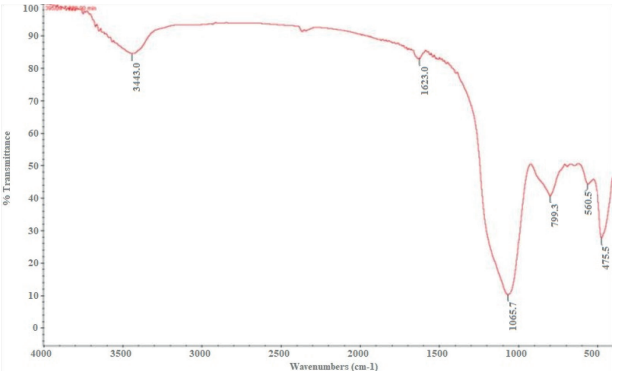

Fig. 7c IR spectrum of thermally treated kaolinite at 600 °C for 90 min
7c. ábra A 600 °C-on 90 percig hőkezelt kaolinit IR spektruma

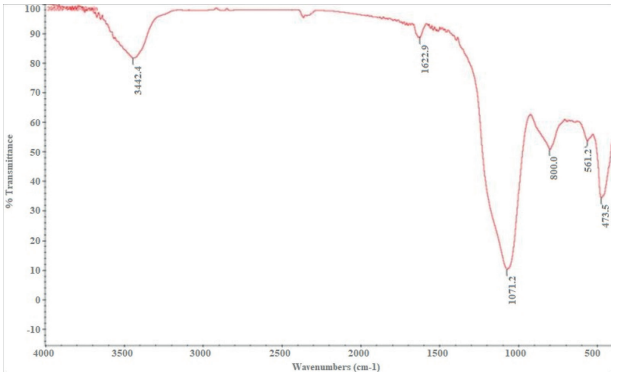

Fig. 7d IR spectrum of thermally treated kaolinite at 800 °C for 90 min
7d. ábra A 800 °C-on 90 percig hőkezelt kaolinit IR spektruma

Gravimetrically, kaolinite samples were heated at different temperatures and times, in order to determine the optimal heating parameters. The mass losses of the pre-and post-heated samples are listed in *Table 2*.

Table 2 shows that the mass loss increases up to 180 min, for heating temperatures from 100 up to 750 °C, while extending heating has no significant change of mass loss. It was found that at temperature 700 and 750 °C and heating period = 90 min., the obtained values for mass loss are the same (11%). From economic point of view and according to international trend to conserve energy, the optimal heating temperature is 700 °C and heating period is 90 min.

The mass losses during heating at different temperatures and the L.O.I obtained by chemical analysis (M_{max}), the degree of

de-hydroxylation calculated via equation (2) are summarized in *Table 3*. It was found that the de-hydroxylation has been achieved to near completion after 90 min. at temperature 700 °C, where the degree of de-hydroxylation (D_{tg}) = 0.99.

Heating period, min.	Temperature, °C										
	100 humidity	200	300	400	500	550	600	650	700	750	800
15	0.47	0.04	0.12	0.17	0.90	2.10	3.65	7.25	9.45	10.50	10.67
30	0.65	0.07	0.15	0.21	1.60	4.65	7.00	9.85	10.60	10.65	10.82
60	1.09	0.11	0.17	0.35	3.15	6.85	9.65	10.50	10.90	11.00	11.15
90	1.11	0.12	0.22	0.51	3.80	8.35	10.05	10.75	11.00	11.24	12.36
120	1.12	0.15	0.29	0.64	3.44	8.75	10.08	11.18	11.58	12.00	12.39
150	1.23	0.21	0.62	0.83	5.32	9.10	10.25	11.00	11.85	11.91	12.41
180	1.35	0.32	0.78	0.95	5.85	9.55	10.34	11.09	11.95	11.97	12.50

Table 2 *The mass losses up to 180 min, for heating temperatures from 100 up to 800 °C*
2. táblázat *180 percig, 100 és 800 °C közötti hőmérsékleten történt hőkezelés során kialakuló tömegveszteség*

Heating period, min.	Temperature, °C										
	100	200	300	400	500	550	600	650	700	750	800
15	-	0.003	0.01	0.014	0.072	0.168	0.292	0.580	0.70	0.84	0.85
30	-	0.006	0.012	0.017	0.128	0.372	0.560	0.788	0.86	0.85	0.87
60	-	0.01	0.014	0.028	0.252	0.548	0.772	0.840	0.98	0.88	0.92
90	-	0.01	0.018	0.041	0.304	0.668	0.804	0.860	0.99	1.00	1.00
120	-	0.012	0.023	0.05	0.392	0.700	0.808	0.877	1.00	1.00	1.00
150	-	0.017	0.05	0.07	0.426	0.728	0.820	0.880	1.00	1.00	1.00
180	-	0.026	0.062	0.076	0.468	0.764	0.827	0.887	1.00	1.00	1.00

Table 3 *The degree of de-hydroxylation*
3. táblázat *A dehidroxiláció mértéke*

According to the peak of kaolinite de-hydroxylation (*Fig. 8*), the transformation of kaolinite to meta kaolinite for the tested samples started at 500 °C and finished around 700 °C, the temperature which was then found to be sufficient for transformation. This temperature is in agreement with the results of previous studies. It was reported that the calcinations temperature must be higher than 700 °C to have a best reactivity of kaolinite [28] and must not exceed 800 °C.

With high temperature, alumina and silica can be re-organized again into new thermodynamically stable compounds (mullite, tridimite, etc) with no reaction with lime hydrate.

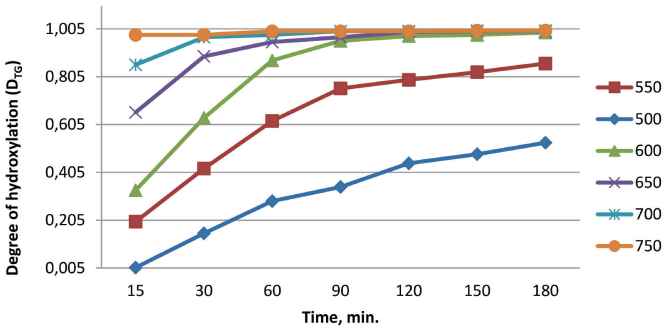

Fig. 8 *The degree of de-hydroxylation at different temperatures*
8. ábra *A dehidroxiláció mértéke különböző hőmérsékleteken*

Optimized heating process can be achieved at 700 °C for 90 min, the activity for pozzolanic meta-kaolinite ranged between 0.65 and 0.74 g $Ca(OH)_2$/g of meta-kaolin, which is in agreement with the previous studies that reported a range of (0.55 - 0.82 g $Ca(OH)_2$ for each gram of meta-kaolinite [11].

4. Conclusions

The produced meta-kaolinite as a source of active silica and alumina was prepared by thermal treatment of local kaolin deposits found in South Sinai, Egypt. The formation of meta-kaolinite was investigated by XRD, IR analyses. The optimum conditions for heating process are temperature 700 °C for 90 minutes to have a best reactivity of kaolinite. The process revealed that the de-hydroxylation degree equal 0.99. The pozzolanic activity was determined by Chappell test, the obtained average value of 0.70 g $Ca(OH)_2$/g meta-kaolinite indicated that the produced meta-kaolinite can be used as an active source for fabrication of geopolymer adsorbent material.

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