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Design and fabrication of cylindrical ceramic crucible as insulator for energy storage systems

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Abstract. The ubiquitous demand for thermal energy in domestic and industrial applications, necessitates the need that heating systems, are effectively insulated, portable and occupy less space. This research focuses on the design and fabrication of a portable crucible for insulating a thermal energy storage system operating at high temperature. The insulating properties of the crucible were also investigated. Two ceramic crucibles, comprising of kaolin, and a mixture of kaolin and sand, were fabricated using the parameters obtained from simulation. When subjected to thermal treatment, the crucible comprising of kaolin and sand structurally failed at 75 °C. However, the crucible comprising of only kaolin remained stable and attained steady state at a temperature of 200 °C. The heating and cooling temperature curves of the kaolin crucibles were plotted and the estimated thermal conductivity of the kaolin crucible was 0.09 W/mK at 200 °C; this is in good agreement with theoretical values of kaolin which range from 0.03 to 0.3 W/mK. The computed thermal diffusivity is 4.9×10^{-8} m²/s, which is much lower when compared with the thermal diffusivity of insulating materials like polystyrene, glass fibre and rock wool. Thus, the rate at which heat diffuses through the crucible is low, making it suitable for insulating a thermal energy storage material operating at 800 °C. The results of this study will facilitate an efficient way of transporting stored thermal energy in portable insulating containers.

1. Introduction

The energy potential of renewable energy sources as energy carriers is instigating efforts towards research and development of sustainable and efficient energy storage/conservation-technologies. According to Zhang *et al.* (2016) [1], the four major goals satisfied by energy storage include providing a balance in the timely and geographical mismatch between demand and supply thereby promoting the integration and generation of renewable energy sources, management of transmission and distribution of grids resulting in stabilization of the energy grid, facilitating the transition to an efficient and more sustainable use of energy with energy storage technologies, thus contributing to a competitive and secure energy supply. Energy storage is an absolutely essential component in energy systems which allows the efficient utilization and conservation of energy from available renewable energy sources.

Currently, many energy storage systems exist of which thermal energy storage systems are the least expensive, having a wide range of applications such as generation of electricity using concentrated solar power (CSP) plants, industrial applications and domestic/building applications such as space heating, hot water supply, etc. [2]. Thermal energy storage (TES) systems employ technologies in which thermal energy is captured from renewable energy sources or recovered from waste heat and stored in materials which have the capacities to store energy with minimal

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losses for later use. The materials selected for storing thermal energy are based on factors such as thermophysical properties, operating temperatures, availability and cost, as well as the type of heat transfer fluid (HTF) and heat exchanger design [3]. TES systems are mainly of three types, each operating on different principles; sensible heat storage (SHS), latent heat storage (LHS) and thermochemical energy storage. In SHS, thermal energy is stored as a result of the temperature gradient in a medium. This heat storage type usually involves no phase change as the temperature of the material simply increases or reduces. However, in LHS systems, phase change occurs in the medium while the temperature remains constant. This storage type usually has a higher energy per volume compared to SHS [4]. Thermochemical heat storage involves the storage of heat in a reversible chemical reaction in which the heat stored is equal to the enthalpy of the reaction. However, developments in thermochemical heat storage is complex and still remains at the laboratory stage. The simplicity, availability of sensible heat materials and lower cost of constructing SHS systems give SHS some advantages over LHS. Also, LHS materials such as molten nitrate salts are limited in their use in high temperature applications such as electric power generation because of their chemical and thermal instabilities at elevated temperatures (above 500 °C) [3,5,6].

Since SHS systems store thermal energy due to temperature difference in the medium, heat loss is unavoidably incurred and can become substantial especially when stored over a long period of time [7]. Consequently, they are typically insulated to prevent energy loss, although these insulating systems are too large to be conveniently transported. For instance, hot water storage systems which are used for domestic hot water supply are usually in large volumes in the range of 500L to several cubic metres (m³) [8]. Other storage systems such as packed-beds (comprising soil, sand, rocks and clay), aquifers and boreholes are stored underground. They usually have no insulation except for those provided at the ground surface and cannot be easily transported due to their location and size. The size constraints of these systems limit their availability and applicability to areas where they can be easily sited. Yet, the ubiquitous demand for thermal energy necessitates that it is made available in systems or containers which are effectively insulated and occupy less space, thus improving the efficiency of the system and its portability. This study focuses on designing and fabricating a novel portable thermal insulating system with locally available material, in which a high temperature SHS medium can be enclosed with no leakage and minimal heat losses over a period of time.

2. Materials and Method

2.1. Crucible Design

2.1.1. Thermal Insulating Material.
The thermal energy storage (TES) system simulated to operate at an elevated temperature (up to 1200 °C), requires a suitable material to withstand the temperature without undergoing thermal stress or chemical degradation [9]. While taking account of the upper temperature limit of the proposed TES, refractory materials were considered for use as the insulating material for the crucibles. Kaolin was selected as the thermal insulator due to its favourable thermophysical properties for energy application as well as its availability in Nigeria. According to Michot et al. (2008) [10], kaolin also known as aluminosilicate ($Al_2Si_2O_5(OH)_4$) can withstand temperatures as high as 1700 °C with thermal conductivity values ranging from 0.03 to 0.3 W/mK. The thermal conductivity of kaolin varies with moisture content, temperature, density and porosity [11,12]. Thus, to increase its porosity and further reduce its thermal conductivity, it was mixed with combustible fillers i.e. rice husks prepared to look like sawdust. It was expected that upon firing the refractory crucible, the fillers will burn off leaving tiny holes. The motionless air in the holes will reduce the large surface area for convection and subsequently improve the insulating property of the crucible. However, the mechanical stability was taken into account and precaution was taken not to increase its porosity beyond an optimal level.

2.1.2. Geometry.
The geometry of a crucible impacts its durability during thermal cycling. Moreover, it affects the convective heat transfer coefficient which is a factor for controlling heat loss to the surrounding fluid. The geometry of the thermal insulator has to be designed such that it maximizes the thermal and mechanical performance of the system. Several geometries were considered for the thermal insulation crucible based on three factors; the effects of force distribution and stress

distribution and the packing efficiency of the geometry. When compared to other geometries, the wall of a spherical-shaped container gave the least surface area (A) and will subsequently experience less force (F) for the same amount of pressure (P) as given in equation (1):

$$P = F/A$$

(1)

Lesser force on the walls of the crucible means better mechanical stability of the whole system under thermal stress or load. The shape of the sphere facilitates better distribution of stress along its walls as there is no concentration of force at a particular point unlike in cubes or cuboids where the edges may experience larger stress and breakdown leading to cracks and leakages in the system. A cylindrical shape will give the next least surface area and will also experience less stress along its walls. However, in terms of packing efficiency, the cube/cuboid is most suitable, with the sphere having the least efficiency. Although the sphere is more durable in terms of stress distribution along its walls, it is difficult to manufacture and cannot be easily stacked due to its shape. Thus, the cylindrical geometry was selected.

2.2. Determination of Thermal Conductivity of Material

The thermal conductivity of the insulating crucible was determined using the hot-wire method. The hot wire method is a transient technique for measuring thermal conductivity and is ideal for measuring the thermal conductivity of materials which typically have low thermal conductivity values such as soil samples and refractories [13,14]. A linear heat source such as thin wire was placed inside the isotropic and homogenous test sample, with the assumption that the heat flow within the sample is a one-dimensional radial heat flow. Constant electric current was passed through the wire and its thermal conductivity was computed from the change in temperature of the material over the distance between the hot wire and the outer surface of the material within a finite time interval. The hot wire setup discussed here mimics the type described by Zhao et al. (2016) [15] and extensively discussed by Antoniadis (2011) [16]. The thermal conductivity of the insulating crucible sample is expressed in equation (2):

$$\Delta T = T(t_2) - T(t_1) = \frac{p}{4\pi k} \ln\left(\frac{t_2}{t_1}\right)$$

(2)

where $T(t_1)$ and $T(t_2)$ are the temperatures at the time intervals, t_1 and t_2 respectively, p is the constant heating power.

2.3. Method of Moulding

Before the crucibles were finally produced, there were four trials in order to obtain the best method of moulding the crucibles. The first crucible was moulded by applying the wet cast method [17], in which two litres of water was mixed with 2 cl sodium silicate (binder). Kaolin powder was added gradually while the mixture was continuously stirred until it attained a slightly uniform thickness. Two cylindrical plastic pipes of diameters 12 and 7 cm and heights 14 and 11 cm respectively, were used as the moulds for the desired shape. The moulding of the crucible involved two setups; A and B. In setup A, at the centre of the 12 cm diameter pipe, another plastic pipe of diameter 7 cm and height 11 cm was placed to create a hollow, and the kaolin mixture was poured in to fill the height of the outer cylinder. Setup B consisted of the 7 cm diameter plastic pipe, which had a candle with a diameter of 3 cm and height of 7 cm placed at its centre and the kaolin mixture was poured in to fill the height of the outer cylinder. The setups were kept under a shade and left to air dry for 7 days.

The second trial involved using the dry press method of moulding [18]. Kaolin pre mixed with water and sodium silicate binder, already wet moulded and dried, was crushed and separated into different grain sizes using the mesh sizes of 5 mm, 2 mm and 1 mm. 19 cl of water was mixed with sodium silicate as a binder and allowed to set for 5 minutes after which 2.05 kg kaolin with grain size <5 mm was added to the solution. Kaolin of grain size <2 mm weighing 0.55 kg was dry-mixed and transferred into the mixture along with another mixture of the same amount of binder and water as earlier stated. 1.35 kg of kaolin of grain size <1 mm was then added to the

mixture and the consistency of the mix was adjusted by adding 7 cl of water. The final mixture was allowed to set for 30 minutes in order to ensure even distribution of water and proper hydration of the mixture after which the mixture was loaded into the compression testing machine (YES-2000, maximum loading capacity of 2000 kN) and pressed with a force of 189 kN. A cylindrical hollow was carved out of the cylindrical kaolin cast using a cylindrical steel of diameter 3.5 mm and height 4 mm. The product was left to air dry for 1 day before being heated overnight in a furnace at a temperature of 70 °C.

In the third trial, the dry press method was employed although the kaolin clay which was moulded consisted of different compositions of sand and sawdust to observe their effect on the thermal and mechanical performance of the final product. Sawdust was added to increase the porosity of the kaolin crucible and consequently reduce its thermal conductivity. Sand was added because it has been reported that silica sand increases refractoriness i.e. the ability of the material to resist melting or burning at high temperatures when fused with the TES. The first sample consisted of a mixture of kaolin and sawdust in the ratio 2: 0.5 by volume, the second mix consisted of kaolin and sand mixed in the ratio 2: 1 by volume and the third sample consisted of the mixture of kaolin, sand and sawdust in the ratio 2: 1: 0.5 by volume. All samples were mixed with 12.5 cl of binder (sodium silicate) and 37.5 cl of water each. The final mixtures were dry pressed in the compression testing machine and the aggregate crushing machine. After pressing, it was difficult to make a hollow in the semi-wet moulded mixtures due to their compositions, thus, they were simply extruded. They were left to air dry for 7 days after which they were gradually heated in the furnace up to a temperature of 900 °C.

In the fourth trial, the refractory was moulded by dry pressing and consisted of two batches with different compositions. The first batch contained just kaolin, while the second contained kaolin and sand mixed in the ratio 3:1 by mass. The process involved mixing 5.4 kg of dry powder-kaolin with 90 cl of water while the second batch which contained a mixture of sand was moulded separately using the same process. Both samples were mixed without any binder. They were left to set for six hours to allow the water distribute evenly in the mixture. The set mixtures were then pressed into cylindrical moulds using the compression testing machine at a pressure of 160 kN and left overnight, after which they were extruded. Both samples were dried in an oven set at temperature 60 °C for three days. After oven drying, a hole of diameter 2.5 cm was drilled in both samples. The height of the cylinder was 13 cm and the depth of the hole was 6.7 cm while the base of the cylinder had a thickness of 3.13 cm.

2.4. Data Collection

The moulded crucibles (kaolin and kaolin/sand mixture crucibles) were subjected to dielectric heating in an 800 W microwave operating at a frequency of 2.45 GHz. The temperature of the cylindrical thermal insulating crucibles along the vertical and angular directions were measured using the 568 contact and infrared temperature gun manufactured by Fluke, at an interval of three minutes, for one and half hours. The temperature was recorded at 0°, 90°, 180° and 270° in the angular direction ϕ and along the length of the cylinder (axial direction, z). The average temperature was computed at the top and base of the crucible. The temperature was limited to 200 °C to prevent the plastic base in the microwave from melting. After heating, temperature readings were taken at 10-minute interval as the crucibles were cooling. Temperature measurements taken at the top of the crucible were recorded as T_u while temperature measurements taken at the base of the crucible were recorded as T_d.

3. Results and discussion

3.1. Cylindrical Ceramic Crucible Fabrication and Analysis

The first trial which involved producing the crucibles using the wet cast method did not give desirable results. There was formation of cracks during the air-drying process of the crucibles due to plastic shrinkage of the mixture. Also, the outer surface of the crucibles did not completely dry because they were covered with the cylindrical plastics which were used as moulds. The cracks formed are depicted in Plates 1a and b. The cracks observed on the surface of the wet cast crucibles

during the drying process are possibly the result of the occurrence of plastic shrinkage. Plastic shrinkage cracking is most likely to occur before the hardening of the clay mixture if the evaporation rate is faster than the rate at which bleed water (i.e the water that rises to the surface as the solid particles in the mix settle) is replaced [19].

In Plate 1b, pores were observed on the surface of the cylinder. The visible pores on the surface of the wet cast are as a result of trapped air in the mix. Fine aggregates such as the kaolin powder used for the mix, tend to trap air bubbles. Although smaller air bubbles help improve the insulating properties of the crucible, larger air bubbles adversely affect the mechanical properties of the crucible as they become weak points during strength tests. Also, due to the fact that the mixture was not compressed, the intermolecular spacing between the grains was larger leading to propagation of cracks. One way to combat this effect is by compression which is achieved by the dry press method of moulding.

The dry press method employed in the second trial yielded much better result than the wet cast method. The compaction pressure was higher, so there was no crack formation and no presence of pores. However, the creation of a hollow in the crucible while it was wet and the extrusion of the wet crucible from the mould was quite difficult. The extra pressure added in creating the hollow while the crucible was wet, created cracks which resulted in re-moulding and retrying the process many times until one crucible was successfully produced. Before the crucible was heated, the inner and outer diameters of the crucible were 6 cm and 15.2 cm, respectively. After being heated, the inner and outer diameters became 8.7 cm and 15.2 cm respectively. For effective insulation of the TES material, thicker insulation walls are required. The second trial failed in this regard because, as soon as the wet mould was air-dried and heated in the microwave, the crucible shrank and the hollow which was created when the crucible was wet became wider, thus reducing the thickness of the insulating material. This means that the hole in the cylinder should be created after it has been heated.

The crucibles produced from the third trial, which had sawdust in them were brittle and chipped off easily. Perhaps, the quantity of sawdust added greatly increased the pores in the mixture such that it affected the mechanical properties of the crucibles. Also, the sawdust did not burn off as expected, possibly due to uneven heating in the furnace. Plates 1c and d present the crucibles from the fourth trial. The height of the crucibles was 0.13 m each, with a radial thickness of 0.125 m. The dry masses of the kaolin crucible, and the kaolin and sand crucible were 3.6 kg and 4.2 kg respectively.

Plate 1: a) Crack observed on setup A; b) Small pores observed on surface of set up B; c) Crucible composed of kaolin/sand mixture; d) Crucible composed of kaolin

3.2. Thermal Treatment of the Crucibles

During heating in the microwave, the crucible containing kaolin-sand mixture cracked at 75 °C. The crucible composed of the mixture structurally failed and this could be as a result of uneven expansion of the constituent materials. Uneven expansion is more likely to occur in non-homogenous mixture of crystalline materials, hence, the formation and propagation of cracks within the crucible. Moreover, the addition of silica sand to kaolin may increase its thermal storage capacity and cause it to act like a thermal energy storage material rather than an insulator. For the purpose of the desired objective, the effect of the addition of sand is undesirable as it considerably increased the mass of the crucible, thus reducing its portability. However, the sample consisting of only kaolin reached steady state at 200 °C in 72 minutes. It took approximately 1.5 h to cool the kaolin crucible to 100 °C in a convective atmosphere of $h = 25$ W/m²K.

In Figure 1, although the initial temperature of the crucible was uniform, it was observed that during heating, there was a difference between the temperature at the top and at the base of the crucible. However, as the crucible approached steady state, the temperature flux becomes lesser and the crucible temperature becomes almost uniform. Similarly, the cooling curve (Figure 2) exhibits the same trend as the crucible cools. This implies that heat is transferred in the axial direction (z-coordinate) which opposes the assumption of only radial heat transfer.
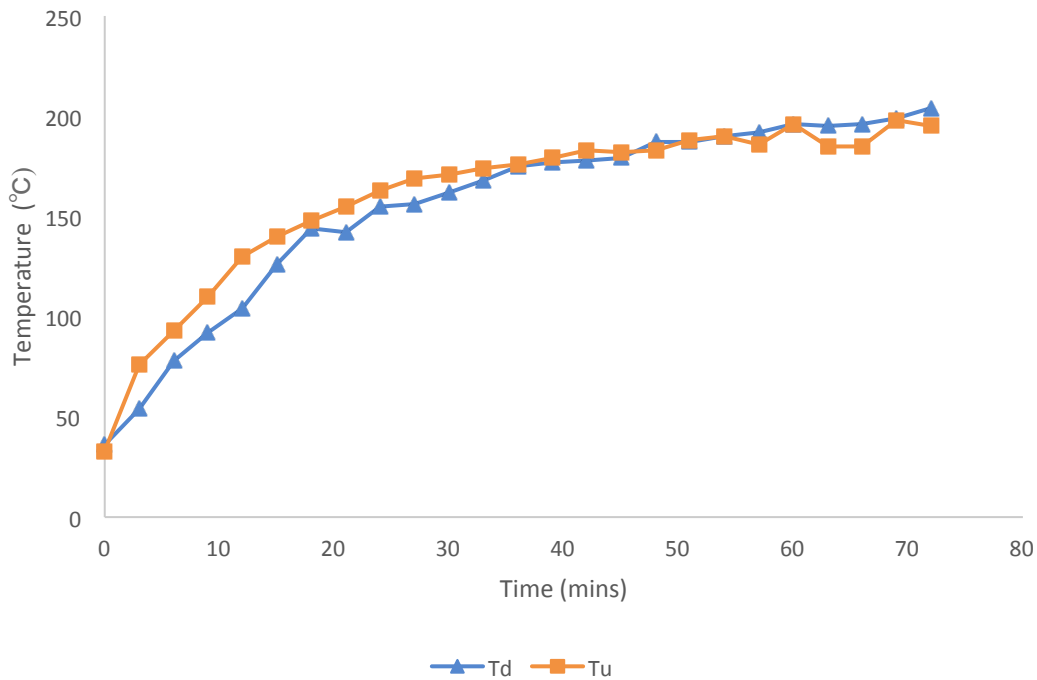
Figure 1: Heating temperature curve of kaolin crucible taken at its top (T_u) and base (T_d)
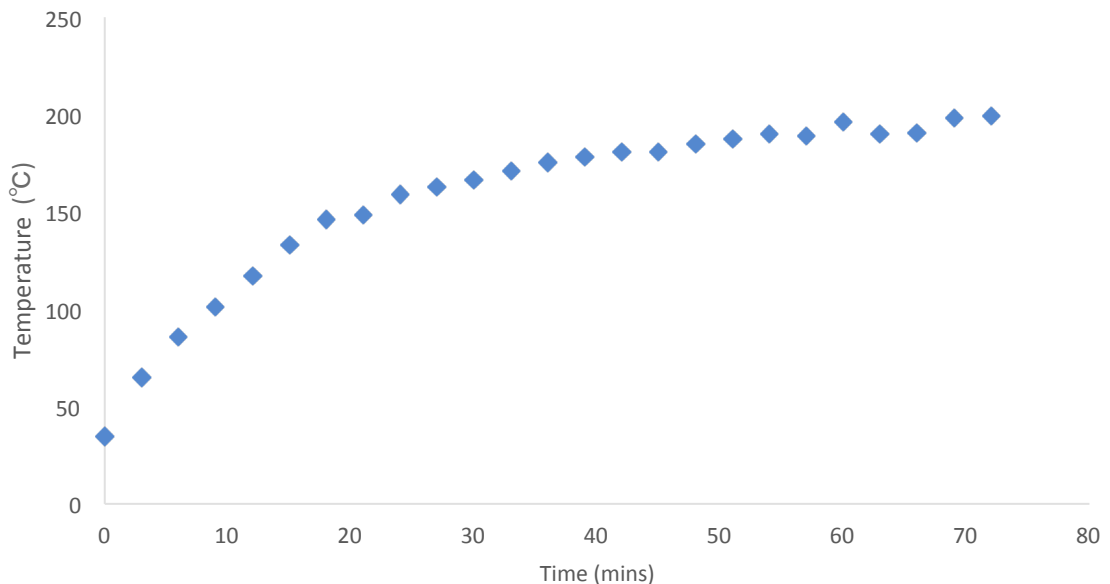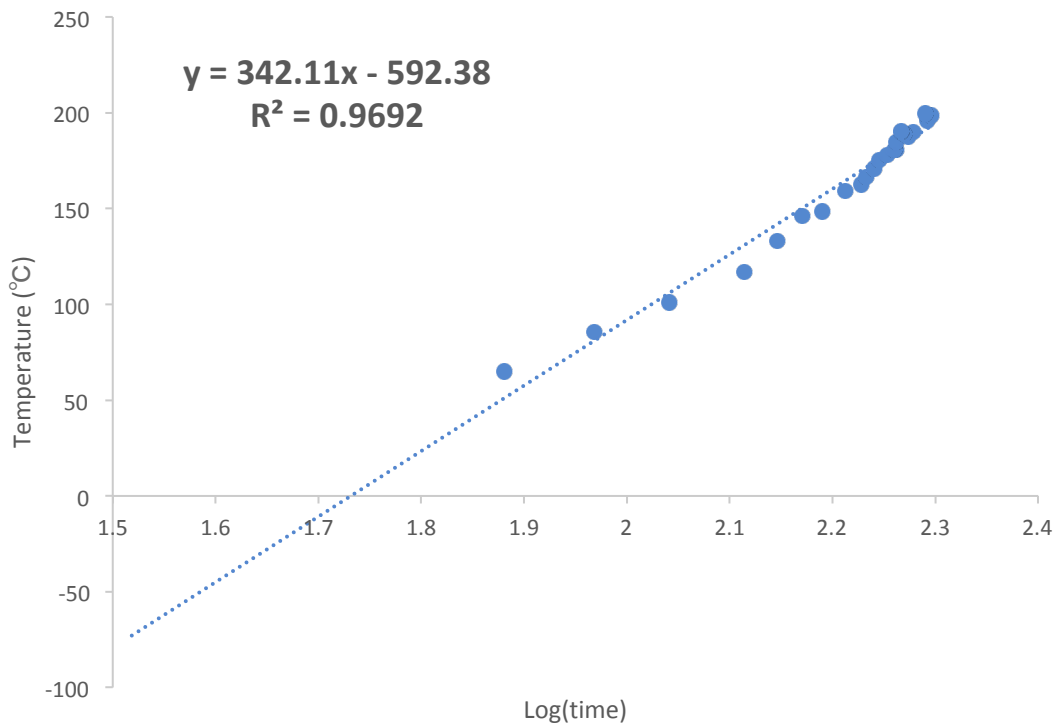

Figure 2: Average heating temperature of the kaolin crucible

The average surface temperature values during heating were plotted against the log of time (Figure 3), to deduce the thermal conductivity of the crucible. Applying equation (2), the slope (342.11) of the plot was used to calculate the thermal conductivity. The thermal conductivity of the crucible which was calculated to be 0.09 W/m K at 200 °C is within the theoretical values of kaolin which range from 0.03 W/mK at room temperature to 0.3 W/mK at 1050 °C [10]. Also, when these values are compared with those of other thermal insulators such as rock wool (0.049 W/mK) and glass fibre (0.034 W/mK), both taken at room temperature, it is found to be within

the standard limit. The temperature curve of the crucible measured at its top and base while cooling is presented in Figure 4. The average cooling temperature is presented in Figure 5.

Although the thermal conductivity varies with temperature, the specific heat is a function of the composition of the material and does not change with temperature. Thermal diffusivity of the crucible was calculated using the deduced thermal conductivity value and the theoretical value of specific heat obtained by Al-Ajlan (2006) [20] as 1009 J/kgK. The computed thermal diffusivity is 4.9×10^{8} m²/s. This value is lower compared to the thermal diffusivity value of some thermal insulators presented by Al-Ajlan (2006) [20]. Thus, the implication is that the rate at which heat diffuses through the crucible is low.

$$y = 342.11x - 592.38$$
$$R^2 = 0.9692$$

Figure 3: Graph of kaolin crucible's average heating temperature against log of time
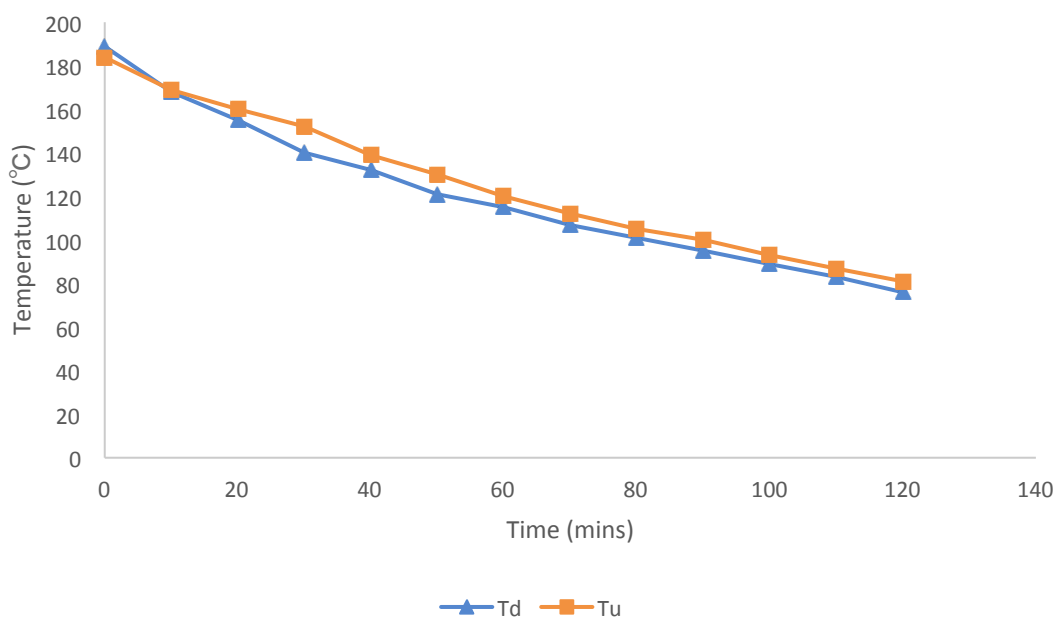
Figure 4: Cooling temperature curve of kaolin crucible taken at its top (T_u) and base (T_d)
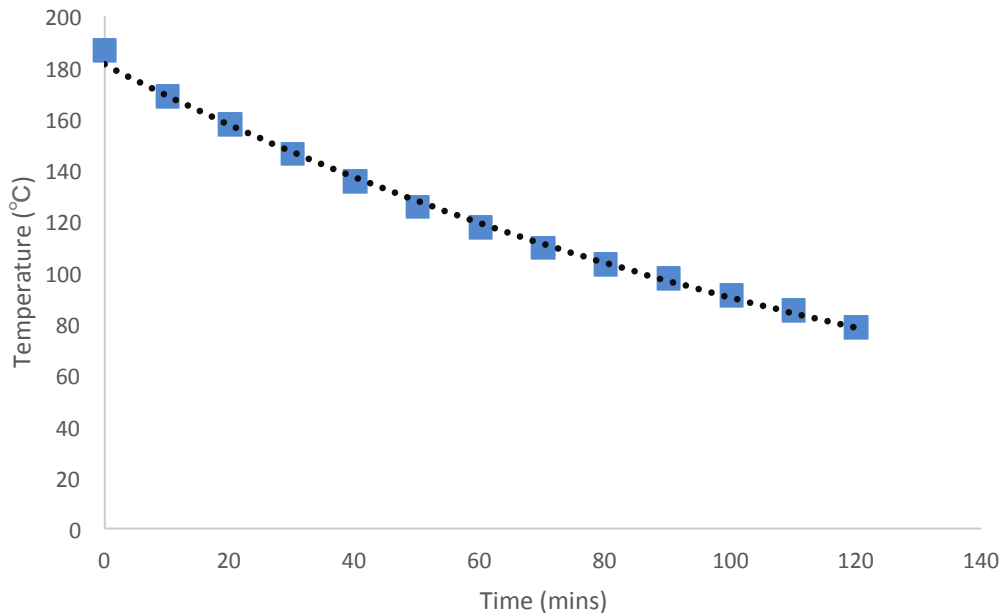
Figure 5: Average cooling temperature of the kaolin crucible

4. Conclusion

Two cylindrical ceramic crucibles which are intended to insulate a thermal energy storage system were successfully designed and fabricated. Based on the methods employed in fabricating the crucibles, the dry press method provided better results. Considering the contents of all crucibles, the crucible comprising only of kaolin performed better under thermal tests conducted. Additionally, the thermal conductivity and the rate at which heat diffuses through the kaolin crucible are very low which make it a good thermal insulator for TES application. Further investigation can be carried out on the operational life of the kaolin crucible through thermal cycling.

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