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Interpretation of Mass Transfer in the Vertical Shaft Kiln (VSK) Process of Cement Manufacture using TGA/DTG Study

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Abstract

The present study is related to the mass transfer occurring in the vertical shaft kiln (VSK) process of cement manufacturing using TGA/DTG study. The TGA/DTG results are compared with the experimental result for varying operating parameters of air flow rates and nodule diameters. The different phases of the mass transfers that occur are explained in terms of TGA/DTG mass loss and experimental mass loss. It is found that TGA/DTG analysis can be effectively used for interpretation of the mass transfer associated with the VSK process.

Keywords: Mass transfer, TGA/DTG, Vertical Shaft Kiln, Cement Manufacture, Packed bed

Introduction

Manufacturing of cement using Vertical Shaft Kiln (VSK) Process is widely used for small scale cement production particularly in developing countries [1]. From the process point of view vertical shaft kiln is a gas-solid counter current, moving bed and chemically reacting system / reactor which is thermodynamically acting as a gas-solid heat exchanger [2, 3]. Manufacture of cement through vertical shaft kiln is made through a process called the black meal process, wherein the fuel, usually low volatile coal or breeze coke is inter-ground with raw material viz. lime stone, clay and additives, if any to form a homogenous powder called the black meal. Green nodules of size 8-12 mm diameter are prepared in a rotating disc nodulizer with spray of approximately 8-10% water and then fed in to the vertical shaft kiln where it is burnt by supply of compressed air from the bottom [4]. Mass Transfer phenomenon is associated along with the heat transfer phenomena in the Vertical Shaft Kiln Process of cement manufacture.

In the present study, the mass transfer phenomena is attempted to be explained from Thermo gravimetric analysis (TGA) and Differential Thermo Gravimetric (DTG) analysis comparing with the macroscopic observation of mass measurement from different experimental runs carried out in a designed and fabricated prototype VSK [4].

Mass transfer process in VSK

Mass transfer is mass in transit as the result of a species concentration difference in a mixture [5]. Just as a temperature gradient constitutes the driving potential for heat transfer, a species concentration gradient in a mixture provides the driving potential for transport of that species. During the VSK process, the first phase of mass transfer is the evaporation of water from the green nodules, with the water vapour coming out from the nodule surface. Next phase of mass transfer is during the burning of the coke breeze, when oxygen of air reacts with carbon and CO_2 comes out. The third phase of mass transfer is during the calcination process when $CaCO_3$ decomposes to CaO and CO_2 where CO_2 comes out from the nodules [2, 3]. In the first and third phase, the heat transfer drives the mass transfer, but in the second phase both heat and mass transfer have simultaneous effect. Inside the individual nodules, there is counter radial movement of air and CO_2.

Materials and methods

It is mentioned that mass transfer study has been carried out using Thermo Gravimetric analysis (TGA) and Differential Thermo Gravimetric (DTG) analysis. Thermo gravimetric is a thermal technique which continuously measures the mass of a sample subjected to a steady increase of temperature in order to quantify reactions involving gaseous emissions [6]. DTG can be obtained from the TGA plot where, first derivative of mass loss with respect to temperature is expressed as DTG i.e. $\frac{d}{dT}$ (Mass change) $= \frac{dm}{dT}$ vs. temperature. The equipment used in the study is Jupiter STA 449 F3 from Netzsch, Germany. Figure 1 shows the equipment.

The sample used for the analysis is the raw mix prepared for the production of green nodule, proportioned as per the mix design i.e. limestone, coke breeze and clay in the ratio of 21.18 : 5.71 : 3.11(all grinded form up to the fineness of 90µ) [4].

Fig. 1: TGA/DTG equipment.

Results and discussion

The first phase mass loss is evaporation of water as shown in the figure 2. It is found to be 5.05% against the DTG peak of 69.9^{0}C and continues up to 250^{0}C. Although theoretically water evaporates at 100^{0}C, the evaporation of combined water takes place up to the temperature range of 200-250^{0}C [7]. During the preparation of green nodules 8-10% of water is used and some water evaporates during the process of sample preparation and hence evaporation mass loss shown by TGA/DTG is 5.05%, which is acceptable.

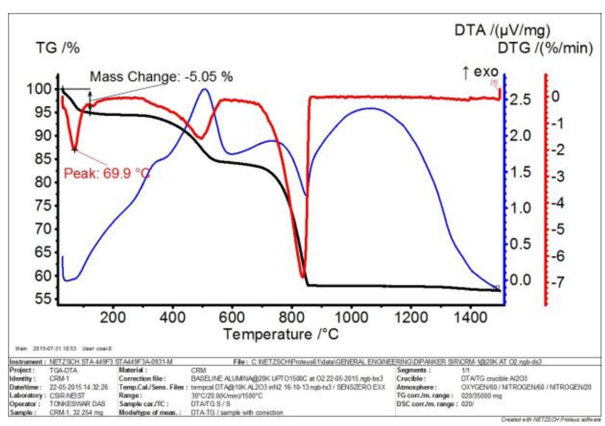

Fig. 2: TGA/DTG plot of mass loss from evaporation of water.

The second phase of mass change which was observed from the thermo gravimetric analysis is 10.65% as shown in the figure 3. It is against the DTG peak of 495.8^{0}C. Chemically in this step coke breeze is combusted to form carbon dioxide as $C + O_2 \rightarrow CO_2$. During this process, the air enters through the pore space of the nodule into its centre and the produced CO_2 also comes out through the same pore spaces.

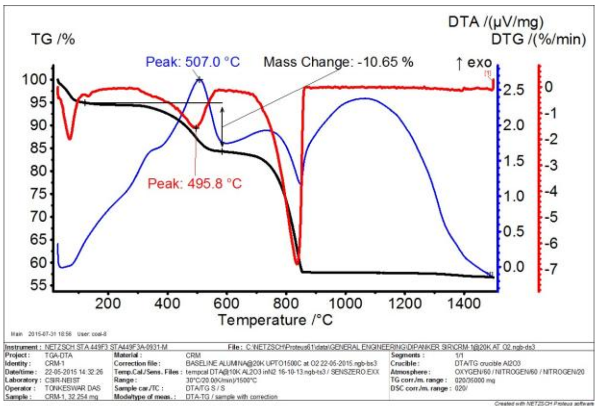

Fig. 3: TGA/DTG plot of mass loss from combustion of coke breeze.

The percentage mass loss from the raw mix design can be calculated as

$$= \frac{3.11\,\text{kg}}{30\,\text{kg}} \times 100 = 10.36\%$$

This value is at close approximation with the TGA/DTG result.

The third and last phase of mass change was observed as 26.43% from the thermo gravimetric curve as shown in figure 4. It is against the DTG peak of 836.0^{0}C. Chemically in this step $CaCO_3$ is dissociated to CaO and CO_2. In this case there is only one way mass transfer, where CO_2 comes out from the surface of individual nodules.

In the raw mix design of 30 kg batch the limestone ($CaCO_3$) present is 21.18 kg and 10% of water is added during nodulization which resulted in a total mass of 33 kg per one batch. Since one mole of $CaCO_3$ produces one mole of CO_2, therefore, 21.18 kg of limestone ($CaCO_3$) is equivalent to 211.8 moles. Thus the amount of CO_2 produced was also 211.8 moles and in terms of mass it was 9319.2 gm = 9.32 kg.

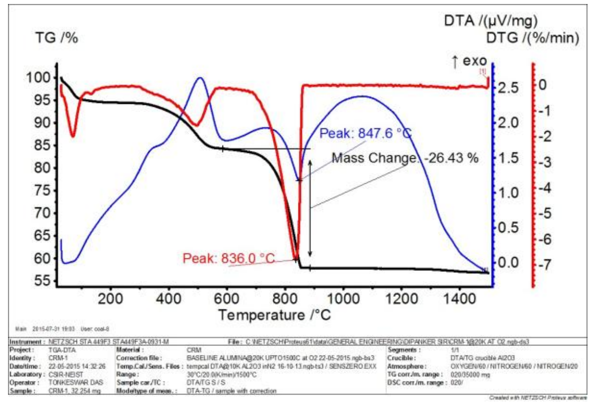

Fig. 4: TGA/DTG plot of mass loss from calcination of limestone.

Therefore, 9.32 kg of CO_2 was evolved from the total mass of 33 kg and hence the mass loss

$$= \frac{9.32\,\text{kg}}{33\,\text{kg}} \times 100 = 28.24\%$$

This value is also in close approximation with the TGA/DTG result.

It is also observed from the TGA graph that after 900^{0}C there is no mass change in the system. After that temperature, only solid-solid reactions take place with no mass change. After 1300^{0}C there occurs the phase transformation process, which again has no mass loss [7].

Figure 5 shows the TGA/DTG plot of total mass loss occurring in the VSK process and it is found that the residual mass is 43.23%.

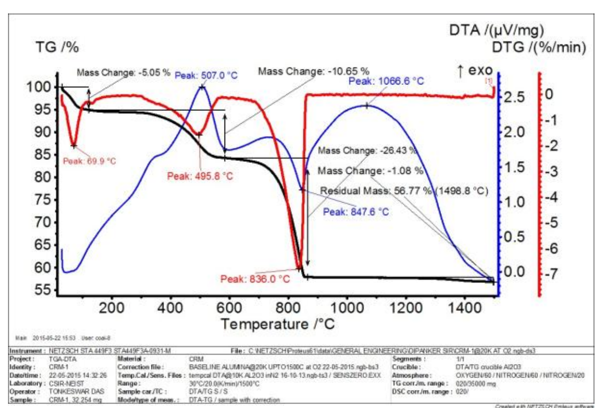

Fig. 5: Total Mass loss observed in the TGA/DTG plot.

Experimentation using the designed VSK was carried out for 6kg and 30 kg batches [4]. The mass loss is measured using digital weight balance for varying nodule sizes (7.02 mm, 8.72 mm and 10.21mm) and for varying air flow rates (2000 litre per minute or lpm, 2500 lpm and 3000 lpm). Table 1 and Table 2 shows the test results for 30 kg batch and 6 kg batch respectively. The obtained results are compared with respect to air flow rates and nodule diameters. (As shown in the figure 6 and 7).

Table 1: Result of 30 kg batch.

Sl. No	Nodule Size (mm)	Air Flow Rate (lpm)	Initial Batch Weight of Green Nodules (kg)	Final Batch Weight of produced clinkers (kg)	% age mass loss (Experimental)	Mass loss from TGA analysis %	% age deviation from TGA result
1		2000	30	17.1	43.00	43.23	0.53
2	7.02	2500	30	17.2	42.67	43.23	1.30
3		3000	30	17.3	42.33	43.23	2.07
4		2000	30	17.4	42.00	43.23	2.85
5	8.72	2500	30	17.5	41.67	43.23	3.62
6		3000	30	17.5	41.67	43.23	3.62
7		2000	30	19	36.67	43.23	15.18
8	10.21	2500	30	20.5	31.67	43.23	26.75
9		3000	30	21.1	29.67	43.23	31.37

The reason behind this deviation (for both the cases) is due to the incomplete mass transfer in the 3rd phase i.e. incomplete conversion of $CaCO_3$ to CaO. This may be accompanied with the incomplete mass loss in second phase i.e. combustion of C to CO_2. In this case the reaction front did not reach the core of the nodules. In the kiln study, it is observed that the maximum temperature did not reach the value of 1450^0C, which indicates the incomplete calcination reaction [4]. The reason for not reaching the temperature of 1450^0C is the higher bed porosity of bigger size nodules, which provides easy path of the air through the packed bed to the chimney exhaust without reacting with the packed bed materials.

Table 2: Result of 6 kg batch.

Sl. No	Nodule Size (mm)	Air Flow Rate (lpm)	Initial Batch Weight of Green Nodules (kg)	Final Batch Weight of produced clinkers (kg)	% age mass loss (Experimental)	Mass loss from TGA analysis %	% age deviation from TGA result
1		2000	6	3.45	42.50	43.23	1.69
2	7.02	2500	6	3.45	42.50	43.23	1.69
3		3000	6	3.46	42.33	43.23	2.07
4		2000	6	3.46	42.33	43.23	2.07
5	8.72	2500	6	3.47	42.17	43.23	2.46
6		3000	6	3.46	42.33	43.23	2.07
7		2000	6	3.85	35.83	43.23	17.11
8	10.21	2500	6	4.15	30.83	43.23	28.68
9		3000	6	4.25	29.17	43.23	32.53

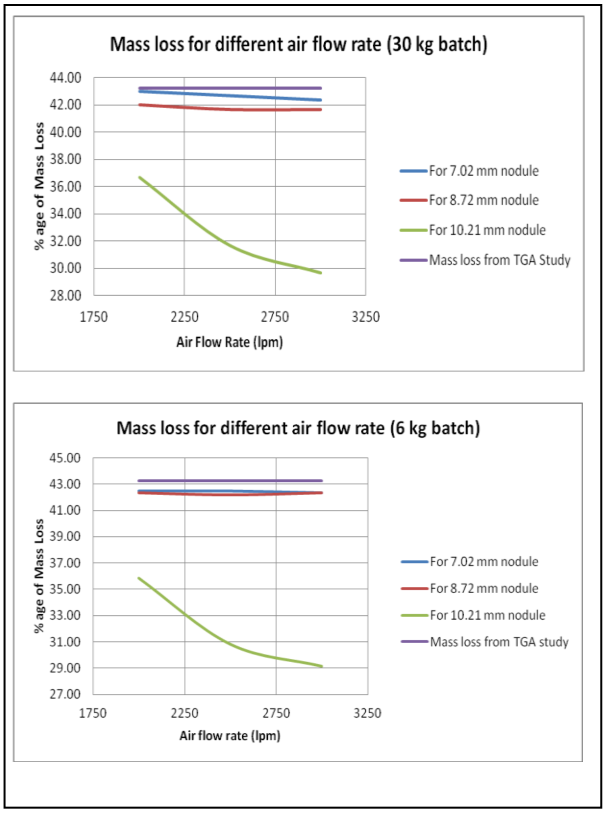

Fig. 6: Mass loss with respect to different air flow rates.

It is observed that in both the cases, the 10.21 diameter nodule deviates highest from the TGA/DTG result.

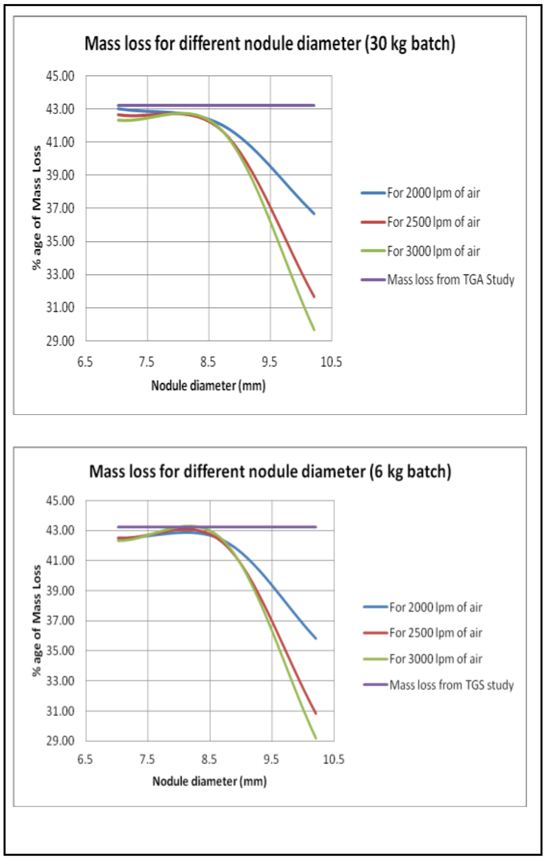

Fig. 7: Mass loss with respect to different nodule diameters.

Conclusions

It can be concluded that the TGA/DTG mass loss is in close approximation with the experimental result of the prototype vertical shaft kiln operation. Mass transfer inside the individual nodules leads to the mass losses occurring in the packed bed and the final mass loss is the additive effect of all the phases of individual mass transfers. It can also be concluded that for bigger size of the nodules used in the packed bed, the process of manufacturing cement is not completed. The inadequate mass transfer inside the individual nodules, due to air channelling through the packed bed leads to incomplete heat generation and thereby none reaching the appropriate calcination temperature. Thus a reliable prediction of mass transfer can be made from TGA/DTG study.

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