TSEFLAR™ – the centrifugal flash reactor for rapid thermal treatment of powdered materials

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Abstract

The concept of the centrifugal thermal activation (CTA) of powdered materials on a rotating solid heat carrier is developed. A compact centrifugal flash reactor has been manufactured and tested. The tested set-up provided: the heating of gibbsite powder to the temperature of 400–900 K with the rate more than 10^3 K/s; the duration of the powder thermal treatment less than 1.5 s; the regulation of the treatment duration by changing the speed of the plate rotation and the shape of the rotating heat carrier; the product output up to 40 dm^3/h; the removal of the extracted steam containing gases and rapid quenching of the thermally activated solid products. The device tested has been used for the thermal activation of commercial aluminium hydroxide Al(OH)$_3$ (gibbsite). The obtained thermally activated product possesses a high reactivity and can be used for the preparation of various alumina-based compounds.
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Keywords: Centrifugal flash reactor; Thermal Treatment; Powdered materials

1. Introduction

Rapid heating of powders followed by their quenching is important in many fields of chemical engineering. For instance, the first stage of preparation of alumina-based supports and catalysts is often performed through the so-called "thermochemical activation (TCA)", i.e. a short-time thermal treatment, of aluminum hydroxide at dehydration temperature, about 580 K, resulting in the formation of a substance with a high reactivity [1]. Phase changes of solid oxygen containing compounds (hydroxides, salts, basic salts, clays, etc.) under heating are mainly determined by the dynamics of two physico-chemical processes. The first one is the removal of labile functional groups (OH$^-$, NO$_3{}^-$, CO$_3{}^-$ and others), the second is the transformation of an initial crystal structure to that of oxide. Under slow heating, these processes are conjugated; therefore an inactive raw material transforms into another substance, a crystalline oxide mainly, which pos-

sesses, as a rule, a low reactivity. Otherwise, when heating is non-equilibrium, with the rate of hundreds degrees per second or more, the formation of a novel quasi-stable amorphous phase can occur [2]. This intermediate phase is characterized usually by a stored energy and thus high chemical reactivity.

Dehydration of crystal aluminum hydroxides or thermal decomposition of aluminum salts under non-equilibrium conditions create the basis of the *low-waste* technologies of the production of active aluminas. When preparing supports, catalysts, sorbents, etc., the use of thermally activated substances as the raw materials allows one to decrease considerably harmful drains and exclude some technological stages: the dissolution of crystal hydroxides, the repeated precipitation, the wash of sediments and others [2].

A widely commercialized method of TCA is based on a dynamic contact of powder particles with either hot exhaust gases or hot granules of support or catalyst [1–3]. However, there are some disadvantages of this approach to the rapid thermal treatment. These are, namely: bad ecology, pollution of the powder with products of the fuel combustion, instability in heating conditions and low efficiency of the heat power utilization resulting in a high energy consumption.

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As an alternative to the existing TCA technologies, we have developed a new approach to the rapid thermal treatment of powders in a so-called centrifugal flash reactor (the Russian abbreviation is "TSEFLAR[TM]"). The TSEFLAR[TM] set-up is simple, compact, environmental benign and provides the more effective heat transfer to the reagent bed with a high rate of the powder particles heating and quenching.

2. The TSEFLAR[TM] design

2.1. Heat carrier

Evidently, the rapid thermal treatment of powder particles is more efficient, with the lower consumption of energy, when the initial powdered substance is contacting with a solid heat carrier, rather than with a gaseous one. Using some easy calculations, it may be shown that the rate of the particle heating to the necessary temperature in the two mentioned cases is essentially different. For providing a simple estimation of the heating rate, we took the next assumptions:

– a particle under heating has two parallel plane sides, one of which is contacted with the hot solid surface; the distance d between the plane sides is the size of a heated particle;
– heat exchange between the hot solid surface and the particle occurs mainly through these sides;
– the particle under heating has initially the temperature $T_0 = 293$ K;
– the temperature of ambient air is equal to that of the solid heat carrier.

The heating rate under the contact with the hot air was estimated for a spherical particle of diameter d. The calculations of the heating time were performed for aluminum hydroxide, $Al(OH)_3$, assuming that its heat capacity = 1.2 J/g K, heat conductivity = 1 W/m K and specific weight = 1.2 g/cm^3. The Table 1 demonstrates the estimated time which is needed for the temperature in the hydroxide particle center to achieve the temperature of the $Al(OH)_3$ dehydration which is known to be equal to 580 K. Parameter T_H in the Table 1 presents the temperature of the heat carrier.

A short duration of the powder particles contact with the heated solid surface can be arranged by centrifugal forces. For example, the powder of the initial substance can be fed into the central part of the hot rotating plate, which is profiled with a broadening to upwards. The centrifugal force ensures the reagent particle movement along the heat carrier surface as well as the steady thermal contact of the surface and the particle until reaching the edge of the plate. Evidently, the residence time will depend on the rate of the plate rotation and the plate' shape as well as on the size of the heated particles, their density, coefficient of friction, temperature, etc.

2.2. The scheme of the "TSEFLAR[TM]" set-up

Fig. 1 illustrates the scheme of the "TSEFLAR[TM]" set-up. The powder of a raw material is falling down from tank 1 through metering unit 2 to hot receiving cone 3 of the rotating plate 4. While moving along the plate on spiral-like trajectory, the heating of the powder particles is in progress. Electrical heating units 5 are disposed in insulated body 6 under the plate. The temperature over the plate is controlled by a set of thermocouples. The cylindrical lateral surface of body 6 is cooled by water through conduits 7. The powder under heating is moving in a narrow cavity between the rotating plate and insulated cover 8.

The time of the reagent residence on the plate, i.e. "contact time", is regulated by the speed of the plate rotation. Centrifugal forces press the powder to the plate surface thus providing the heat transfer under direct contact of the particles with hot metal. In our opinion, this way of thermal treatment is much more efficient than at the common TCA-process based on the gas heat carriers. As shown above, the rate of heating by a hot gas is almost of an order of magnitude lower.

Coming off the plate, the powder continues a spiral movement down the inner cylindrical surface of the cooling unit 9 that is filled with circulated water. The contact with the cold wall ensures an efficient quenching of the thermally activated products. Falling down, the powder passes through turning cavities 10 to tanks 11.

Some technical characteristics of the manufactured and tested device are the following:

Mass of the total set-up (kg)	1500
Diameter of the set-up body (mm)	1400
Height of the set-up (mm)	1880
Diameter of the rotating plate (mm)	1000
Speed of the plate rotation	90–250 turns per min
Volume of tank 1 (dm^3)	40
Product output, up to	40 dm^3 per hour
Temperature of the rotating plate, up to	1000 K
Supplied electrical power (kW)	20–45

2.3. Studies on the movement of the gibbsite powder over the rotating plate

The movement of the $Al(OH)_3$ powder over the rotating plate surface has been studied using the video technique. The gibbsite product from the Pikalevo Silica Complex (Rus-

Table 1
The calculated values of heating time

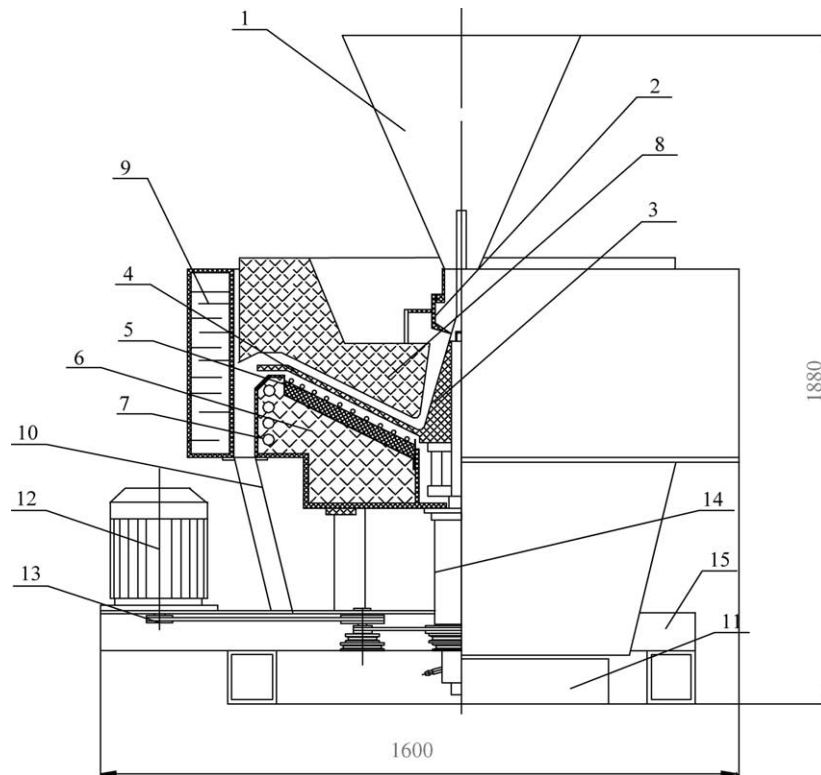| d (mm)	Time of heating to 580 K (s)					
	$T_H = 673$ K		$T_H = 873$ K		$T_H = 1073$ K	
	Gaseous	Solid	Gaseous	Solid	Gaseous	Solid
0.04	0.049	0.005	0.017	0.002	0.010	0.001
0.08	0.152	0.018	0.054	0.008	0.032	0.005
0.12	0.239	0.041	0.104	0.017	0.062	0.011
0.15	0.422	0.064	0.149	0.026	0.089	0.017

Fig. 1. The scheme of the TSEFLAR^{TM} set-up. 1 – tank for raw material, 2 – metering unit, 3 – receiving cone of the rotating plate, 4 – the rotating plate, 5 – heaters, 6 – body, 7 – conduit for water, 8 – cover, 9 – cooling unit, 10 – turning cavity, 11 – tank for the thermally treated product, 12 – electric drive, 13 – belt drive, 14 – bearings body, 15 – frame.

sia) was used as the raw material. The initial powder was dried and dispersed by a set of sieves into the fractions with $d = 0$–0.038, 0.038–0.053, 0.053–0.071, 0.071–0.106 and 0.106–0.150 mm. A video camera was fixed from above the opened rotating plate (without cover 8). The movement of a small amount (2–3 g) of dispersed gibbsite started from receiving cone 3 was shooting with the sequence frequency 25 Hz. The temperature T_p of the plate and speed N of its rotation were varied from 290 to 700 K and from 90 to 250 turns per min, correspondingly.

To do experiments with the video recording, several different versions of the rotating plate were manufactured and tested: with the plane, conic, truncated spherical and special curvilinear surface. The last configuration of the plate was designed to provide the movement of a particle with the constant acceleration. The profile of this plate can be determined when taking into account the forces that influence a particle movement: gravity, centrifugal force and friction. One can easily calculate that the corresponding profile is determined by a correlation expression

$$r = \frac{a + g(k + \sin \alpha)}{\omega^2 (1 - k \sin \alpha)}$$

where α is the angle between horizontal and tangent to the plate surface on the distance r from the axis, a is

the acceleration value, k is the coefficient of friction, $g = 9.81$ m/s^2 and $\omega = 2\pi N$, where N is the speed of the plate rotation. The diameter D of all plates was 0.5 m; two plates, conic and truncated spherical, with $D = 1$ m were also used.

Fig. 2 represents an example of the sequence for the gibbsite powder movement over the conic plate. An analysis of the recorded movies has been done using Corel Draw 11 (Corel Photo-Paint 11), Windows Movie Maker, MathCAD 2001. The residence time, τ, increases while N and d values are decreasing, so the minimal value $\tau = 0.24$ s was achieved when $N = 250$ turns per min, $d = 0.106$–0.150 mm for the plane plate with $D = 0.5$ m and $T_p = 290$ K, while the maximum value $\tau = 1.44$ s was achieved when $N = 100$ turns per min, $d = 0.53$–0.71 mm for the conic plate with $D = 1$ m and $T_p = 290$ K. Note, here the N and d values at which the powder particles are braking and "sticking" to the truncated spherical plate according to non-overcoming gravity are not considered. Additionally, it has been found that the rise of the plate temperature to 700 K leads to a small (10–20%) decrease in τ.

Fig. 3 demonstrates some features of the particles movement along the plates of different shapes. It is found that the use of the plate of the "constant acceleration" allows one to achieve the maximal residence of the powder on the surface of the rotating solid heat carrier.

Fig. 2. An illustration of the gibbsite powder movement over the rotating conic plate (the sequence of the pictures with the time delays 0.12 s).

3. Thermal treatment of Al(OH)$_3$

The set-up described in Section 2.2 has been used to provide the thermal treatment of commercial gibbsite produced in the Pikalevo Silica Complex and Achinsk Alumina Complex (Russia). The conic plate with $D = 1$ m, as the simplest one in its manufacturing, has been used as the rotating heat carrier. The treatment has been performed under temperature $T = 600$–900 K at $\tau = 0.5$–1.4 s.

The products of the gibbsite treatment were investigated by BET, X-ray and thermal analysis, electron mi-

croscopy, IR and NMR spectroscopy. It has been found that the initial gibbsite substance transforms into a quasi-stable state, which is called below as the "centrifugally thermally activated" (CTA) product. The phase composition of the product under $T > 700$ K and $\tau \sim 1.4$ s includes gibbsite Al(OH)$_3$ (0–20%), boehmite AlOOH (0–10%), pseudoboehmite AlOOH (0–20%) and X-ray amorphous phase Al$_2$O$_3 \cdot n$H$_2$O, where $n = 0.4$–2.0 (up to 100%). The thermal treatment of the initial powder results in a decrease of its water content from 34.6 to 5–15% and an increase of the powder specific surface area from 2–5 to 100–300 m^2/g be-
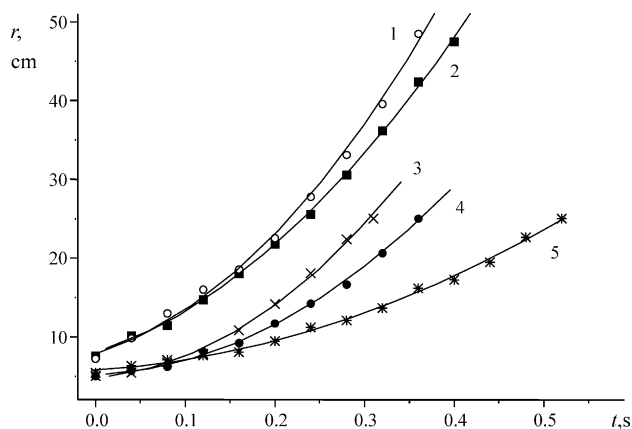
Fig. 3. The recorded movement of the gibbsite particles along the rotating plates of different shapes. r is the distance between the "spot" of particles and the axis of the plate rotation, t is the time. The speed of the rotation $N = 220$ turns per min, the particles size $d = 0.045 \pm 0.007$ mm. 1 – conic plate (with diameter $D = 1$ m), 2 spherical plate ($D = 1$ m), 3 – plane plate ($D = 0.5$ m), 4 – conic plate ($D = 0.5$ m), 5 – plate of the "constant acceleration" ($D = 0.5$ m).

cause of the formation of a microporous system. The particular parameters of the treated powder depend on the conditions of the activation such as the temperature of the rotating plate, the contact time, the size of the particles, the rate of the powder feed, the initial temperature of the powder as well as the pressure of the "steam + air" mixture inside a specially added unit for the steam removal. The net electrical energy consumption under $T = 800$ K appeared to be about 7 kJ/g of Al(OH)$_3$. A detailed information on the physico-chemical properties of the CTA product and its reactivity is presented in this issue by L.A. Isupova et al.

The main parameters of the CTA process are easily controlled and the main physico-chemical properties of the products are well reproduced. The CTA product has a high reactivity in the rehydration process and thus can be considered as a promising starting material for the preparation of various α-, γ- and η-alumina-based compounds including catalysts and catalyst carriers.

4. Conclusion

The device described in this paper can be used for the rapid thermal processing of a large variety of solid dispersed materials as well as for the synthesis of some chemical products. The tested set-up provides: the heating of powders to the temperature 400–900 K with the rate more than 10^3 K/s; the time of the thermal treatment less than 1.5 s; the possibility of the regulation of the treatment duration by changing the speed of rotation and the shape of the rotating solid heat carrier; the regulated product output, up to 40 dm^3/h; the removal of the extracted steam containing gases as well as the rapid quenching of solid products. The main merits of the TSEFLAR^{TM} are the following:

– its compactness and simple design;
– the provision of no environment pollution, since the hot gaseous products of the fuel combustion are not used as a source of heat;
– the properties of the obtained products of the thermal treatment are well reproduced;
– the short contact of the powdered reagent with the rotating heat carrier will not lead to the appearance of additional impurities in the activated products;
– the product output and the energy consumption of the tested set-up are suitable for an industrial coping of the device.

Acknowledgements

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