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Comparison on the drying efficiency of through circulation convective drying and vacuum contact drying of aspirin powder and aspirin agglomerates

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The drying efficiency of through circulation convective drying and vacuum contact drying of aspirin powder and aspirin agglomerates was investigated at initial moisture content of 28 wt.%, temperature of 60°C (temperature of drying gas for through circulation convective drying and heating jacket temperature for vacuum contact drying) and air flow of 12 L/min for through circulation convective drying mode. Through circulation convective drying shows shorter drying time than vacuum contact drying with a final moisture content of 0 wt.% for both the two samples under investigation. Constant heating rate period was obvious in through circulation convective drying but absent in vacuum contact drying. However, for the drying behaviour of aspirin agglomerates, the constant rate period was observed to be within a very short period of time i.e. less than 5 hours in contrast to that of aspirin powder.

Keywords: Drying, moisture, convective, contact and aspirin

1. Introduction

Drying basically means the reduction or elimination of moisture content from a product through heat transfer processes to attain acceptable levels (Ratti, 2001; Keith *et al*., 2005). Drying is a very significant unit operation in the pharmaceutical industry. Even more significant when the final product (such as active pharmaceutical ingredients) characteristics can have effect on the product performance and the levels of residual diluents is seen as a regulatory requirement (Murugesan *et al*., 2011). Drying perhaps have the largest application compared to other units' operation in the industry. Sometimes even a purely mechanical industrial processes could need drying of the product prior to making it set for the market. To date, there is a very poor fundamental understanding of drying of organic materials due to limited research work that has been conducted on it (Gerdner, 1971; Geddes, 2008). An understanding of the drying behaviour of organic materials is significant to taking control over morphology and size distinction of the material and certifying reproducibility of results industrially. So far, most proceeding research on drying focused on inorganic materials (Geddes, 2008), as they are more simply defined and evade the cumulative number of variables present like the morphology of the particles, which often leads to poor industrial applications and processing as well as influence the production of the final products both physically and chemically. This perspective establishes the bases for this study. Consequently, the study aimed at comparing the drying efficiency of two different mode of drying (vacuum contact drying and through circulation convective drying) of two selected organic materials (aspirin powder and aspirin agglomerates) while in contact with solvent using an agitated filter dryer. However, agitation will not be considered in the present study.

Numerous methods are used to transfer heat during drying; sometimes the methods to be used derived benefit on weather moisture to be removed is on the surface of the solid or inside the solid. The most widely used methods of heat transfer for drying are: (1) convection, where heat is transfer from a hot gas or heated air in contact to the material to be dried; (2) conduction, where the material to be dried is in direct contact with the hot/heated surface. Contact dryers have become very popular in the pharmaceutical industry because of their enhanced containment (Sahni and Chaudhuri, 2013), (3) Radiation, where the material to be dried is in view of hot gas or hot surface and; the last but not the least generation of heat within the material to be dried by microwave heating or dielectric heating (Roper, 2006). The method used for heat transfer to the material to be dried is the most significant difference between drying methods (Robert, 2012). The selection of drying method and appropriate dryer in the

pharmaceutical industry depends predominantly on the characteristics of the material to be dried and the scale of production (i.e. small or large scale of production) for instance when non-sphere shaped solids product are required like needles or flakes contact drying is employed (Keey, 1992).

Drying normally consists of three mechanisms: jacket warm up period; which is a short period taken for the system to heat up toward the aim set point; Constant rate period, which is characterize by a constant drying rate as well as speedy vaporization of the solvent that has saturated the surface of the solid transpires during this period; and lastly, Falling rate period, during which drying rate start to drop due to less solvent in the surface of the solid (McCabe *et al*, 2005). The constant and the falling rate period can be detected by determining the temperature of the bed as a function of time (Waananen *et al*, 1993).

Drying is a very complex process with consequences like over drying and snowballing. To date, the impact of drying on the final product is not properly understood (Kukura *et al*, 2001; Adiya and Atiku, 2019). This study specifically, thus, aim at comparing the drying efficiency of through circulation convective drying and vacuum contact drying of aspirin powder and aspirin agglomerates, so as to find out which is more suitable for the drying of the promising active pharmaceutical ingredients (aspirin powder and aspirin agglomerates).

2. Materials and Methods

2.1 Research Materials

Aspirin powder and aspirin agglomerates (aspirin cluster) were chosen for this study because aspirin is one of the major active pharmaceutical ingredients. Another logic behind the choice of the samples is that the aspirin crystals have a well-defined morphology. Therefore, any changes in the size or the shape of the crystals during the drying process could be easily detected and analysed after drying.

Aspirin was discovered in the 18th century when willow and its extracts were used for relieving various pains. This helped in the identification of the active components of willow when their chemical structures were recognized as salicilin, salicylic acid and acetylsalicylic acid. The later were subsequently used in various clinical trials. In 1971, however, the exact pharmacology of aspirin was clarified when aspirin was shown to obstruct with the synthesis of prostaglandins (Mahdi 2006).

2.2 Sample Preparation

1 kg of the dried sample (Aspirin powder or aspirin agglomerates) is weighed, and 1 kg of distilled water measured; the 1kg of the dried sample and 1 kg of water are both mixed into a beaker to generate homogeneous slurry used as feed.

2.3 Running of the AFD procedure

After cleaning of the AFD with water, then acetone before each experimental run, the slurry prepared is charge into the feed port and then the feed port outlet is locked. Nitrogen is pump into the vessel to pressure it up to 1 bar. The outlet valve is open to carry out the filtration process under the driving force of gravity and nitrogen pressure. In this process, the weight of filtrate was recorded. However, in the present study we are only interested in the drying technology. After filtration, the vessel is pressurized up to 2 bars, the three-way valve is switched from filtration to drying position and the drying process is carried out using hot nitrogen gas. A condenser with a flask is placed at the downstream to collect the vapor. A coolant running through the condenser was set to - 5$^{\circ}C$ to cool the condensate. The drying process of the slurry usually take 15-16 hours. By opening the lid, the dry product is collected, and the vessel is cleaned using water and acetone. Exactly the same procedure is used for the vacuum contact drying except that instead of hot nitrogen gas, vacuum is used in the drying process.

Data obtained from the lab view (programmed and installed by SCAPE, University of Leeds) for further processing of results include; filtration rate, temperatures distribution of the four thermal couples in the bed, inlet and outlet humidity of the slurry, heating band temperature, and inlet and outlet temperature all over range of time recorded automatically from the lab view.

3. Results and Discussion

3.1 Aspirin powder

Figure 1 depicts results comparing the efficiency of through circulation convective drying and vacuum contact drying of aspirin powder at initial moisture content of 28 wt.%, temperature of 60^{o}C (temperature of drying gas for through circulation convective drying and heating jacket temperature for vacuum contact drying) and air flow of 12 L/min for the through circulation convective drying. The final moisture content was 0 wt.% for both mode of drying. However, it was observed that through circulation convective

drying has shorter drying time than vacuum contact drying. Equilibrium moisture content is reached in 29 hours in through circulation convective drying while it takes 55 hours to achieve it in vacuum contact drying as shown in figure 1(a). The later is because through circulation convective drying has much bigger heat transfer area than vacuum contact drying. The constant heating rate period is apparent in through circulation convective drying as depicted in figure 1(b), this is in contrast with vacuum contact drying. This observed phenomenon might be attributed to the fact that the movement of water is basically diffusion controlled in vacuum contact drying of aspirin. Overall, the drying rate of through circulation convective drying is faster than that of vacuum contact drying as shown in figure 1(b).

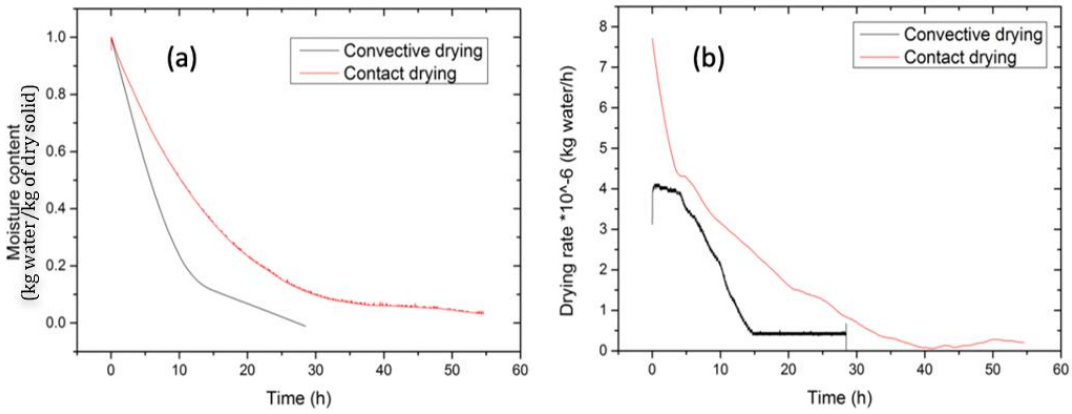

Figure 1. (a) Moisture content against time (b) Drying rate against time

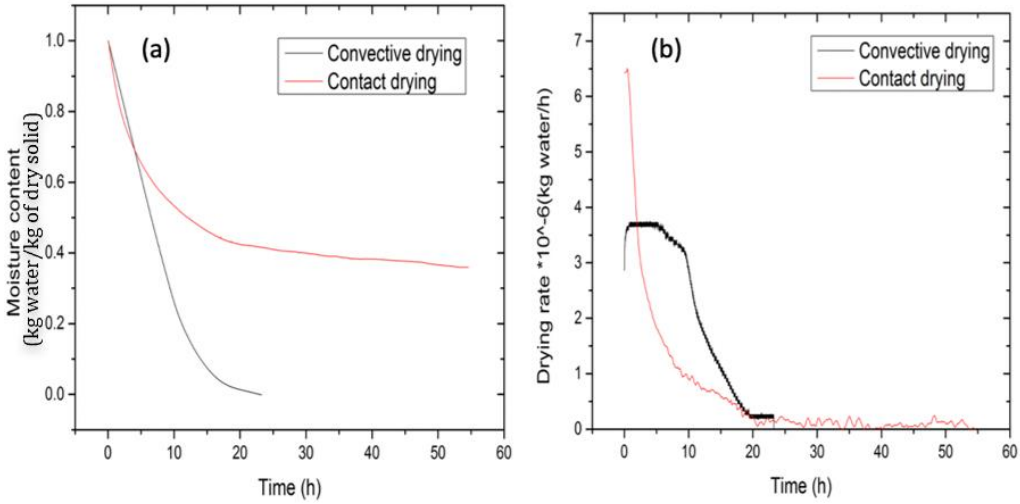

Figure 2. (a) Moisture content against time (b) Drying rate against time

3.2 Aspirin agglomerates

The effectiveness of the two mode of drying is slightly different for aspirin agglomerates but negligible. Final moisture content of 0 wt.% was achieved in through circulation convective drying in contrast to 0.35 wt.% in vacuum contact drying. The drying time of aspirin agglomerates shows similar trend as aspirin powder. That is, the through circulation convective mode of drying has shorter drying time than the vacuum contact drying as shown in figure 2(a), with approximately over 30 hours difference in the drying time. Equilibrium moisture content was achieved in 23 hours in through circulation convective drying while it takes 55 hours to be reached in vacuum contact drying. Figure 1(b) indicate the constant heating rate period clearly in through circulation convective drying but was in a short period of time i.e. less than 5 hours. The constant heating rate period was absent in vacuum contact drying because the movement of water is most likely diffusion controlled in vacuum contact drying of aspirin as mention earlier. In general, the drying rate trend is same as that of aspirin powder.

Direct comparison of the present study with previous studies is not feasible because of differences in either the mode of drying use or samples chosen for the studies. Most drying research, if not all focused on inorganic

materials and/or samples for reasons mention previously in the introduction section. The few that studied organic materials and/or samples used a different drying technology compared to the one used in the present study. However, numerous studies on drying has be conducted using aspirin and other active pharmaceutical ingredients similar to aspirin. For example, (Chua *et al,* 2019) investigated the 'characterisation of the convective hot air drying (convective) and vacuum microwave drying of cassia alata: antioxidant activity, essential oil volatile composition and quality studies." (Adiya and Atiku, 2019) investigated the drying behaviour of salicylic acid, aspirin agglomerates and aspirin powder using a developed model and Thermo Gravimetric Analysis (TGA). (Gohel *et al*, 2004) study the formulation design and optimization of mouth dissolve tablets of Nimesulide using vacuum drying technique. (McLoughlin *et al*, 2003) wrote a paper on 'Microwave-Vacuum Drying of Pharmaceutical Powders'. In 2001, (Kardum *et al,* 2001) investigated the comparative analysis of convective, vacuum and microwave drying of chlorpropamide.

4. Conclusion

It was found that through circulation convective drying is more effective than vacuum contact drying for the drying of the two samples under investigation. This is further validated by the final moisture content of 0 wt.% obtained in the drying of both aspirin powder and aspirin agglomerates and the shorter drying time of through circulation convective drying compared to the vacuum contact drying. Approximately over 25 hours difference was observed in the drying of aspirin powder and over 30 hours difference in drying of aspirin agglomerates between the two modes of drying.

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Conflict of interest

The authors declare no conflict of interest.

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