VACUUM CONTACT DRYING OF PHARMACEUTICAL POWDERS WITH AND WITHOUT AGITATION: A COMPARISON

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ABSTRACT

The drying efficiency of vacuum contact drying of aspirin agglomerates and aspirin powder with and without agitation was compared using an agitated filter dryer (AFD). The aim of the study is to compare the drying efficiency and impact of vacuum contact drying with and without agitation of the samples under investigation. Operating conditions was at 15 wt. % initial moisture content, heating jacket temperature of 75 ℃ and agitation speed of 30 rpm. It was found that vacuum contact drying with agitation is more efficient in the drying of aspirin agglomerates and aspirin powder than vacuum contact drying without agitation. About 49% decrease in drying time was observed in the drying of aspirin agglomerates with agitation compared to without agitation and 64% decrease in the drying time of aspirin powder when agitation was applied, in contrast to without agitation. Thus, equilibrium moisture content was also reached in vacuum contact drying with agitation in a much faster rate than in vacuum contact drying without agitation at exactly same operating conditions.

Keywords: *Drying, vacuum, contact, agitation and aspirin*

INTRODUCTION

Reduction or elimination of moisture content to reach acceptable levels is called drying. Drying is one of the most vital unit operation in the pharmaceutical industry [1-2]. It also has the broadest application in other industry. Even purely mechanical industrial processes might need drying of the product before setting to market. Perhaps around 1 .5 to 2% of the total annual investment in new equipment in the chemical process industries is used for the installation of drying equipment [3].

Vacuum contact drying is an indirect method of eliminating moisture from solid by used of heat, such that the heat transfer channel is detached from the material to be dried by a metal wall [4]. Heat transfer is principally by conduction across the metal wall; hence, these units are also referred to as conductive dryers. Although over 85% of the industrial dryers are through circulation convective dryers [5], vacuum contact dryers give great thermal proficiency and have good environmental and financial benefits than through circulation convective dryers [6-8]. Some of the most significant advantages of vacuum contact dryers are; numerous energy resources can be used, and it has less energy consumption; thus, operational cost will be low. The case is different with direct dryers that normally used fuel or natural gas. Another benefit of vacuum contact dryer (indirect dryers) over direct dryers is that they can be operated in continuous or batch mode, moreover vacuum contact dryers are ideal in the production of active pharmaceuticals ingredient (API). There is also an increase in the desirability of vacuum contact dryers for industrial used because they can be used for wide variety of solids processing like slurries, filter cakes or wet granules. The major challenge with vacuum contact dryers is difficulty in designing and engineering them [7]. Contact dryers are normally desired for heat sensitive materials such as pharmaceutical, foods and other biomaterials. Despite the obvious industrial significant of vacuum contact dryers, it's still the least understood, the correlation for the forecasting of drying manners in vacuum contact dryers of a specific geometry are not available due to the fact that numerous physical phenomena happen at the same time [9-11].

This paper focuses and aim on comparing the drying efficiency of vacuum contact drying of aspirin agglomerate and aspirin powder using an agitated filter dryer (AFD) with and without agitation. An AFD can be described as that dryer that performs both filtration and drying in a single unit operation [12]. Combining more than one single operation within single set of equipment is very important, especially due to decrease in material handling and material lost due to transportation. AFD is used to dry most materials in pharmaceutical and fine chemical industries, either in slurries or pastes form at a reasonable low operating cost because labor requirements are small. It offers the most economic form of non-continues drying for a set of batch size [13]. The unique feature of an agitated filter dryer is the multipurpose agitator. The agitator does numerous operations through movement in axes both parallel and perpendicular to the shaft. AFD can be made-up of materials like stainless steel, mild steel and mild steel with rubber lining as service required. Recently, agitated filter dryers have been reported to be made-up of polypropylene fiber-reinforce plastic (PPFRP). An AFD is often designed as an enclosed and sealed cylindrical shell integrated with an agitator that moves in both parallel and perpendicular axes. The cylindrical shell is usually integrated with heater jacket that is used to control and set the desired temperature of the dryer. Operation is either under pressure or vacuum to desiccate the filter cake. Some advantages of AFD include but are not limited to: very high solvent recovery; inert gas flow rate can be control; insignificant contamination of the cake; pressure or vacuum filtration applicable; considerable saving in manpower; solvents are in closed system, thus no toxic vapor released in the atmosphere, maintaining personal and environmental safety and heat transfer surfaces can be provided to maintain filtration temperature [14-16].

MATERIALS AND METHOD

Research materials

Aspirin agglomerates and aspirin powder were used in this research study as aspirin is one of the major active pharmaceutical ingredients.

Sample Preparation

1 kg of the dried sample i.e. aspirin agglomerates or aspirin powder (depending on sample under investigation) is weighed, and 1 kg of distilled water measured; both are mixed in a beaker to make a homogenous slurry used as feed.

Running of the agitated filter dryer (AFD) procedure

Detailed description of the operational procedure and description of the agitated filter dryer (AFD) and experimental procedure can be found in our previous study Adiya and Atiku, 2020 [14]. However, it is worth noting that in the present study, agitator was used while running vacuum contact drying with agitation at 30 rpm speed.

RESULTS AND DISCUSSION

Drying time is shorter in vacuum contact drying with agitation than without agitation as shown in figure 1. Aspirin agglomerates take about 73 hours approximately to reach equilibrium moisture content with agitation while it takes approximately 148 hours to reach equilibrium moisture content without agitation. The observed phenomenon is relatively not surprising, because mass transfer is from the whole wet cake and heat transfer is from heating jacket to the wet cake. This makes the wet cake to dry layer by layer as heat transfer occurs in the absence of agitation. Thus, the slow drying time in vacuum contact drying without agitation can be explained by the manner the drying process occurs i.e layer by layer in the absence of agitation. Conductivity of the stainless-steel vessel is higher than that of the wet cake. Agitation expose more wet particles to heat transfer area (stainless steel vessel wall), which results in rapid drying of the material. Agitation also develops heat and mass transfer rates by improving the mixing of particles inside the dryer and the particle regeneration rate at the heat transfer surface. As a result, the rate of dehydration at the free surface is increased, leading to high drying rates and a shorter drying time in vacuum contact drying with agitation compared to without agitation.

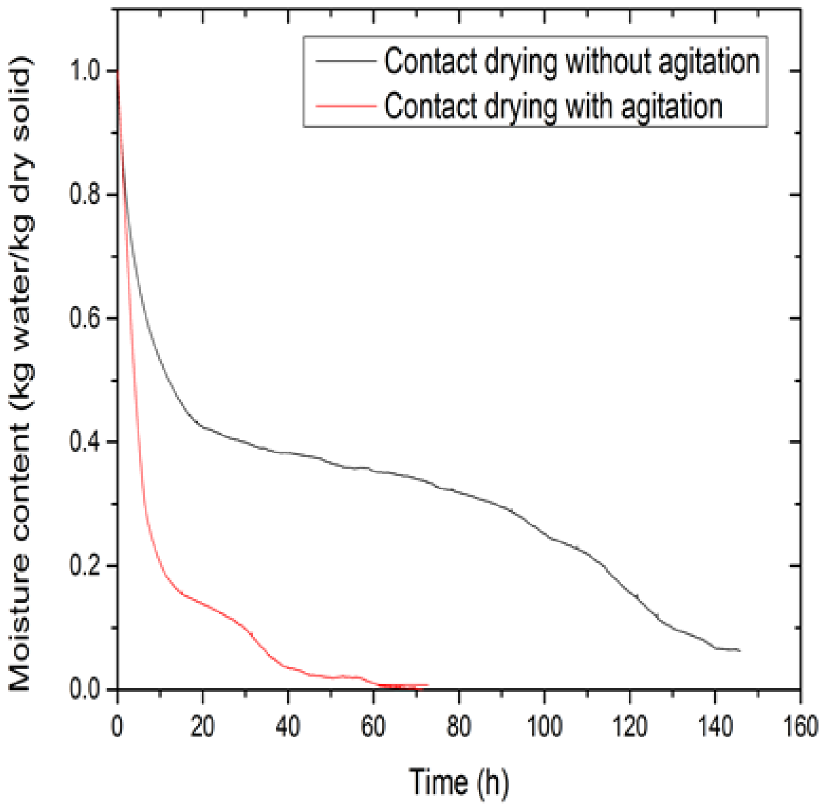

Figure 1: Results of aspirin agglomerates; moisture content against time at 15 wt. % initial moisture content, 75 °C and agitation speed of 30 rpm

Aspirin powder depict similar drying behaviour as aspirin agglomerates as shown in figure 2. It takes aspirin powder about 35 hours roughly to reached equilibrium moisture content with agitation while it takes about 55 hours to reach it without agitation at exactly same operating conditions. However, it is worth noting that aspirin powder has shorter drying time than aspirin agglomerates. i.e the drying of aspirin powder is faster than drying of aspirin agglomerates at exactly same operating conditions both with and without agitation. This behaviour is attributed to the difference in pore size and pore volume of aspirin powder and aspirin agglomerates [17]. The later also explain the observe phenomenon in the final moisture content of the experimental runs depicted in table 1. The effect of agitation reported in the present study is in good agreement with previous studies reported by Lekhal et al., 2004 [18] in his paper titled "The effect of agitated drying on the morphology of l-threonine (needle-like) crystals".

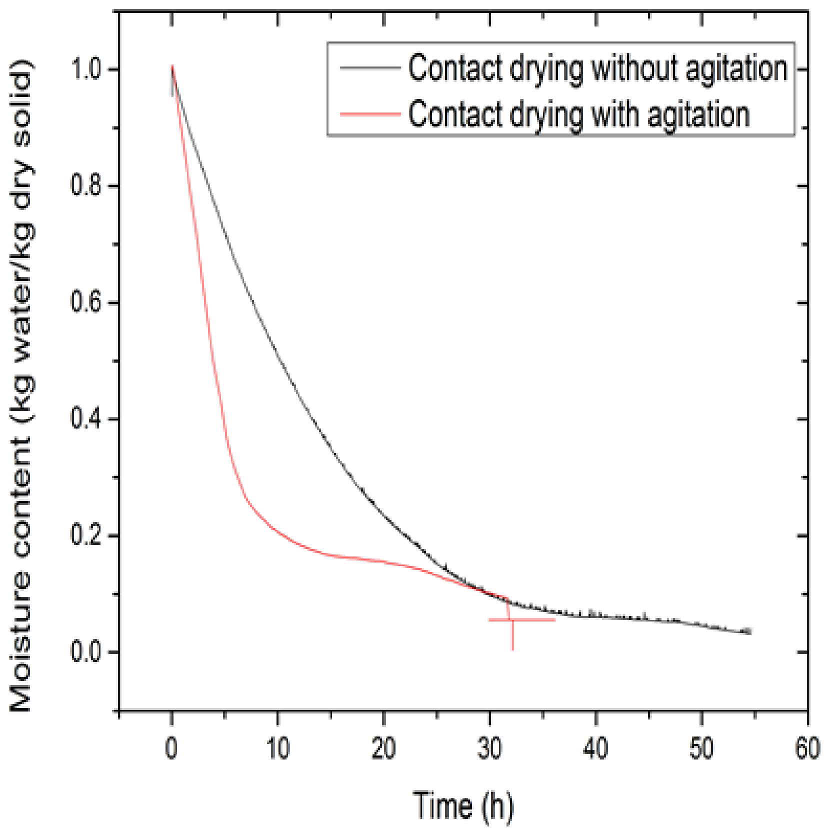

Figure 2: Results of aspirin powder; moisture content against time at 15 wt. % initial moisture content, 75 °C and agitation speed of 30 rpm

Table 1 summarises the conditions of the experimental runs conducted. The table shows values from the initial moisture content to final moisture content as well as the heating jacket temperature, agitation speed and material under investigation. As mention previously, from the results obtained it can be said that vacuum contact drying with agitation is more efficient (with regards to heat transfer, drying time, drying rate, and final moisture content etc.) than vacuum contact drying without agitation. However, drying with agitation is a matter of compromise between its obvious advantages and disadvantages. This because previous studies such as Lekhal et al., 2003 [19] proved that two physical phenomenom (attrition and agglomeratinon) continuously compete during agitated drying. The later was also in good agreemment with the conclusion of Adiya and Mustafa, 2021 [20] in their paper tittled "effect of vacuum contact agitated drying on particle size distribution of aspirin powder and aspirin agglomerates". Kougoulos *et al*, 2011 [21] reported that the effect of agitated drying cause deformation of fibrous crystal habit gotten from the crystallisation causing the formation of irregular granules. Thus, the choise of drying with agitation is a matter compromise.

Table 1: Summary of experimental runs

Run	**Material**	**Initial moisture content (wt. %)**	**Temp. Of heating jacket (℃)**	**Agitation speed (rpm)**	**Final moisture content (wt. %)**
1	Aspirin agglomerates	15	75	N/A	1.9
2	Aspirin agglomerates	15	75	30	0.2
3	Aspirin powder	15	75	N/A	0.0
4	Aspirin powder	15	75	30	0.0

CONCLUSIONS

Although, the choise of drying with agitation is a matter compromise between it advantages and disadvantages, vacuum contact drying with agitation is more efficient in the drying of aspirin powder and aspirin agglomerates than vacuum contact drying without agitation. With regards to drying behavior of materials under investigation, aspirin powder depicts shorter drying time than aspirin agglomerates at exactly same operating conditions.

ACKNOWLEDGMENT

Dr. Tariq Mahmud and Dr. Wei Li are gratefully acknowledged for their guidance and support during this research study.

DECLARATION

Conflict of interest: The authors declare no conflict of interest.

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