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Research Article

Reducing Time of Crystals Formation in Crystallizer using a Hydrofoil as an Impeller and Air Bubbling through the Mixture

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Abstract

Crystallization is one of the most important unit operation in Pharmaceutical industries. Many industries use this equipment in order to obtain the product in crystals for and supply it to the tablet manufacturing companies. We received a problem statement from Granules India Ltd. stating that the cycle time required for crystallization of paracetamol is taking nearly 6hrs for completion. Our task was to reduce the cycle time of the crystallization process. It consisted of 3 stages, mixing time, cooling time and crystals formation time. The temperatures went on decreasing after each stage. We suggested the use of a hydrofoil instead of an anchor and passing air bubbles. Based on the literature survey we concluded that use of hydrofoil reduces cycle time and faster crystallization with the help of air bubbling. Our focus is to study the implementation of hydrofoil and air bubbling in various other crystallizers such batch cooling crystallization, anti-solvent crystallization etc.

Keywords: *crystallization, hydrofoil, air bubbling, batch cooling crystallization*

1. Introduction

Many Pharmaceutical companies use the crystallization process as one of its major process. Since many medicines are based on the crystals formed. Generation of crystals is very important in the crystallizer. The time required for crystals formation is very critical because the entire production of the pharmaceutical industry is based on it. Since most of these industries carry out batch cooling crystallizer, anti-solvent crystallizer or sometimes both.

In general these crystallizer make use of an anchor, paddle or turbine depending on the viscosity of the mixture in the crystallizer. The major disadvantage of these anchors, they make use of more power and tend to form vortex in the crystallizer, despite of baffles present in the crystallizer reactor tank. That doesn't help much in reducing the cycle time. Cycle time refers to the complete time period from the feed entering the crystallizer reactor until its discharge takes place completely.

These big reactors generally don't provide the required surface area needed for the faster formation of crystals. Since the anchor speed developed initially while mixing the turbulence created in the reactor restricts the formations of crystals initial. Therefore it takes time for crystals growth and hence increasing the cycle time.

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For all these problems, considering all the possible factors, using the hydrofoil (preferably used Maxflo W) as a impeller along with suitable bubbling process will efficiently increase the crystals formation rate (based only on the literature study, no experimental data). This study might help the researchers to further work on the following assumptions made in this paper.

2. Use of Air bubbling

Based on the study of effect of ultrasonic frequency on cooling crystallization of paracetamol (Jordens J *et al*, 2014), the data said that the introduction of ultrasonic waves had reduced the time for the formation of crystals by increasing the nucleation rate.

There is no exact mechanism of the ultrasonic nucleation as of now, but Louisnard *et al*. proposed the hypotheses of segregation. This states that the solute and the crystal precursors are segregated, during a very short time, by the large acceleration of the cavitation bubble at the end of the collapse. Therefore, very high levels of supersaturation are created momentarily which enhances nucleation (Jordens J *et al*, 2014).

The major disadvantage of this process is that the components tend to degrade, even that was considered in the experiment and a suitable frequency was suggested for minimum degradation.

Due to this degradation, there is high possibility of components in the crystallizer reactor to form different compounds other than expected since the degradation

leads to formation of various ions of the mixture components, it might be very dangerous if used for tablet development without proper verification of the chemical composition after crystallization.

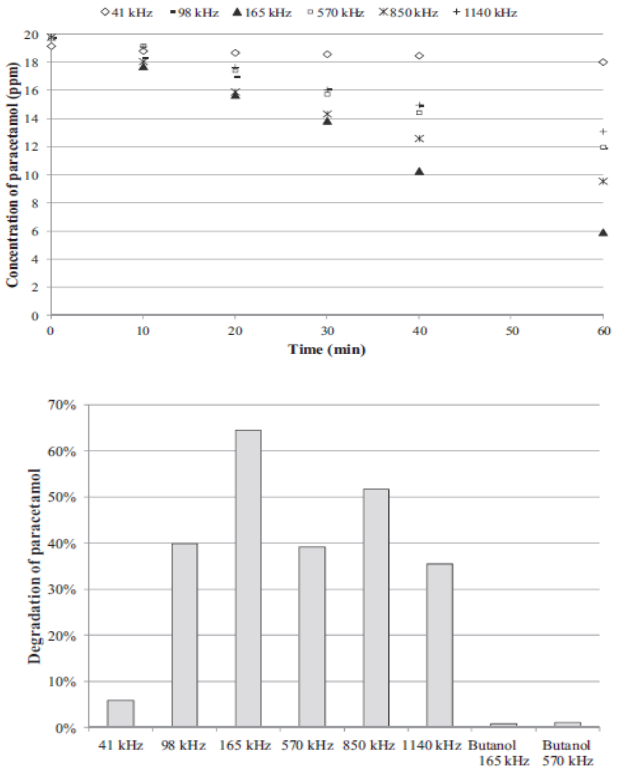


Fig.1,2 (Jordens J *et al*, 2014)

From this concept arose the concept of bubbles. While sending the ultrasonic frequency, there lead to the formation of bubbles and inside the bubble the components disintegrate due to higher frequencies.

Capturing the concept of bubbles from here, instead of bubbles formation due to ultrasonic, we considered the bubbles due to air or nitrogen gas. Preferable nitrogen gas because of its inert nature it will not react with the components in the crystallizer reactor.

There could be two different ways to explain the acceleration of crystallization by air bubbling:

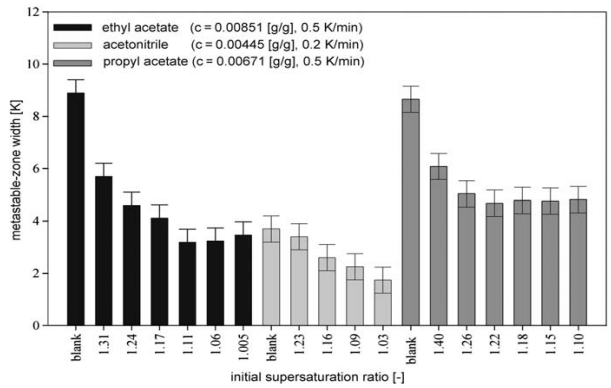


Fig.3. Impact of air bubbling on metastable-zone width for different material systems in comparison to blank conditions

Firstly, the possibility of evaporating solvent into the bubble can assist nucleation by forming an interfacial area around the bubble with a locally higher supersaturation ratio as in the bulk. As a result the driving force of crystallization increases.

Secondly, the gas-bubble surfaces form nucleation sites, which act like foreign particles, and thus, lessen the nucleation energy needed to cross the critical cluster radius (Wohlgemuth K *et al*, 2009).

There are still few limitations for this to take place. These conditions must be followed before sending these air bubbles in the reactor

1) The speed of the impeller must be low, so that the bubbles do not burst because of fast speed impeller.
2) The flow of the mixture must be laminar, turbulence is not preferred for bubbles formation as well as crystals formation.
3) Temperature should be around 40°C - 25°C in case of batch cooling crystallizer.

Another important aspect to considered is the size of the bubbles. Based on the size study can be done for checking which size is preferable for more crystals formation. We made an assumption regarding the size of bubbles, it states that smaller the size of bubble and faster injection across the feed more will be the crystals formation compared to the bigger size bubbles. Only after the required experiments done we can conclude any result.

3. Use of Hydrofoil as impeller

Latest technology developed in the turbines field, generally these hydrofoils were being used in ships, submarines for providing thrust in the seas. It is based on the airfoils used in the airplanes.

Company named 'Chemineer' has been developing these hydrofoils as use for impellers in the reactors. These cannot be simply fabricated like other impellers, advanced laser technology is used for it fabrication.

In general the hydrofoils are used as high speed vehicles, but there are special types of hydrofoils, such as:

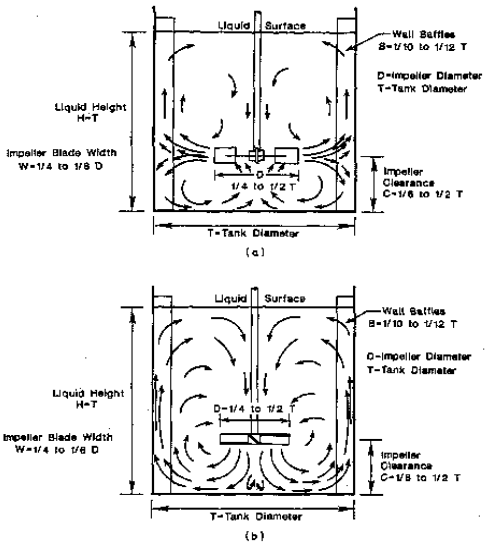
Advantages of Maxflo W Hydrofoil

- Excellent performance in abrasive solids suspension, liquid-solid-gas and boiling or near-boiling applications

- 10% more pumping than the Maxflo T means retrofits with no decrease in performance
- High-solidity blade design translates into improved mass transfer over other high efficiency designs
- It has very low power number, hence it consumes very less power than the traditional anchor.

One of the main reason for high cycle time and less rate of nucleation is due to vortex formation cause due to usage of anchor.

These hydrofoils are specially designed to avoid these vortex formations. These are designed based on the impeller flow pattern. Anchor is based on radial flow pattern but hydrofoil is based on axial flow pattern, hence the formation of vortex is completely eliminated since we are not operating at high speeds.



Axial flow in the crystallizer plays a very important role by not allowing vortex formation. In the crystallization process at we consider three stages (taking the example of paracetamol production):

1) Mixing stage:
   The feed is charged into the crystallizer where the temperature is maintained about 85°C to 90°C in the reactor. Continuous stirring takes place at 70rpm. Here the vortex formation takes place when an anchor is used, which can be minimized with the help of hydrofoil.
   It will further enhance the mixing rate but may initially reduce the nucleation rate. Since the nucleation rate is better at low speed of impeller.
2) Cooling process:
   The reactor is circulated with room temperature water for gradually decreasing the temperature or else the crystals will lead to formation of lumps which is not preferred. Temperature is reduced from 70°C to 40°C.
   Here by placing the speed to around 50rpm to 55rpm along with nitrogen bubbling will further enhance the nucleation rate.
   These conditions of low speed hydrofoil and lowering temperature are very much suitable for bubbles passage through the content in the reactor without getting damaged and hence providing better crystals formation and thereby reducing the crystals formation time and ultimately reducing the cycle time of the process.
3) Crystals formation Process:
   In this section, the rate of crystals formation increases further by sending chilled water at 6°C for reducing the temperature from 40°C to 25°C but traditional use of anchor requires 2hrs to complete the process. Introduction of an hydrofoil and nitrogen bubbling expected reduction time is 1hr.

Another important point to noticed is the speed variations to be considered, generally industries prefer constant speed of the impeller because of which the process is taking lot of time to complete.

Speed variations should be taken care off, when to apply more speed and when to apply less depending on the required rate of crystallization and temperature conditions.

Conclusions

1) This paper is based on providing idea for doing research experimentally for various crystallizers using hydrofoil and nitrogen bubbling.
2) Based on the literature survey, we are concluding that the usage of hydrofoil is beneficial for reducing cycle time in the crystallizer.
3) There are various papers made on using solvents as crystallization medium in anti-solvent crystallizer, experiments can be done using hydrofoil and nitrogen bubbling in these crystallizers.
4) Similarly, various additives are being used for crystallization; there is possibility of further increase in nucleation rate using hydrofoil and nitrogen bubbling.
5) Once experimentally proved, many industries will be benefited from this idea, hence increasing their production rates.
6) There is huge scope of development of crystallizer using hydrofoils and air bubbling.
7) So far, the gassing (air bubbling) technology in crystallization and especially the nucleation process due to gassing have not been investigated extensively like ultrasound (Wohlgemuth K *et al*, 2009).

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