CHEMICAL ENGINEERING TRANSACTIONS
VOL. 49, 2016
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet
Guest Editors: Enrico Bardone, Marco Bravi, Tajalli Keshavarz

ISBN 978-88-95608-40-2; **ISSN** 2283-9216
DOI: 10.3303/CET1649020

Production of Instant Rice Protein Concentrate by Rotating Pulsed Fluidized Bed Agglomeration using Hydrolyzed Collagen Solution as Binder

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The rice protein has raised interest due to its unique nutritional and nutraceutical properties. Rice protein concentrate (RPC) is often used as a food or pharmaceutical ingredient in powder form, however, it presents fine particles and poor instant properties. This work aims to produce an instant and nutritional powder through agglomeration of RPC using a rotating pulsed fluidized bed (RPFB). Aqueous solution of hydrolyzed collagen HC (10 % and 30 % w/w) was used as a binder at flow rate of 1.5 mL/min and pressure of 80 kPa. The air temperature was at 65 ºC. The influence of the binder concentration on the particle size and quality of the product was investigated. The agglomeration was stopped when 120 mL of binder was atomized, after that, the product was dried. The atomization of HC solutions provides the agglomeration, suggesting that HC can be a powerful binder for RPC agglomeration. The evolution of the particle growth was detected by Parsum probe. The binder concentration presented strong influence on the increase of the particle size. Higher binder concentration resulted in granules with median diameter 6 times larger than those of raw RPC, while a 4 time increase was achieved using lower binder concentration. Agglomerated particles had higher protein content, presenting free-flow improvement and lower wetting time. The RPFB was useful to fluidize the RPC particles and to perform the agglomeration, resulting in an instant powder with desirable moisture content. The powder produced presented a high potential to be used as an ingredient in food or pharmaceutical formulations.

1. Introduction

The rice protein has raised interest especially of the food and pharmaceutical industries due to its unique nutritional value (rich in essential amino acids) and nutraceutical properties (Saunders, 1990). This protein is gluten and dairy free which make it a hypoallergenic ingredient (Helm and Burks 1996). The rice protein concentrate (RPC) powder presents fine particles and poor instant properties, limiting its use. In the food industry, the agglomeration processes are performed to the production of instant foods, which are wetted quickly when placed on liquid surface (Hogekamp and Schubert, 2003). The particle enlargement provided by agglomeration improves the handling properties, such as flowability and density, facilitating the transport and storage conditions (Iveson et al., 2001). Conventional fluidized beds are widely used for wet agglomeration. However, pulsed fluidized beds improve the fluidization of fine and cohesive particles (Dacanal and Menegalli, 2010). This process is carried out by atomization of a liquid binder onto the moving particles. The hot airflow causes the evaporation of the liquid binder and the drying of the particles (Tan et al., 2006). The use of a suitable binder is essential to not modify the nutritional value of the final product. Hydrolyzed collagen (HC) is a product of enzymatic hydrolysis of collagen and it consists in a set of amino acids peptides (Walrand et al., 2008). The HC can be a powerful binder for RPC agglomeration, providing the non-use of adjuvants substances like carbohydrates in the production of instant protein.
The monitoring of the particle size during the agglomeration is important, since it is possible to set the stopping point of the process according to the particle size desired. Several studies have reported that spatial filter velocimetry (SFV) technique is an efficient way to monitor in-line particle size in agglomeration process,

Please cite this article as: Andreola K., Da Silva C., Taranto O., 2016, Production of instant rice protein concentrate by rotating pulsed fluidized bed agglomeration using hydrolyzed collagen solution as binder, Chemical Engineering Transactions, 49, 115-120
DOI: 10.3303/CET1649020

using Parsum probe (Silva and Taranto, 2015; Burggraeve et al., 2010). The aim of this work was to study the production of an instant and nutritional powder by agglomeration of RPC using a rotating pulsed fluidized bed (RPFB) and hydrolyzed collagen as binder. The evolution of particle growth was monitored using a Parsum probe. The influence of binder concentration on the particle size and quality of the product was evaluated.

2. Materials and Methods

2.1 Materials

Commercial rice protein concentrated RPC (Axiom Foods, USA courtesy Gramkow, Brazil) was used as raw material for the agglomeration experiments. Aqueous solution of hydrolyzed collagen (Gelita, Brazil) at two different concentrations 10 and 30 % (w/w) and at room temperature (± 27 °C) was used as liquid binder.

2.2 Experimental system

Experiments were performed in a RPFB, equipment used by Andreola et al. (2015). To promote the fluidizing air pulsation, a rotating disk with an opening (60°) was installed bellow the air distributor plate. An electrical motor provides the rotation of the disk. The fluidizing airflow was supplied by an air blower (WEG, 7.5 HP). The airflow rate is calculated by using an orifice plate, measuring the pressure drop across the plate and the pressure upstream of the plate. The air temperature is maintained by an electrical heater, controlled by a PID controller (NOVUS, N1200) and monitored by thermoresistances (Pt-100). Two thermo-hygrometers (NOVUS, RHT-XS) were used to monitor the relative humidity and air dry bulb temperature, located at the inlet and outlet of the equipment. The entrained particles were collected by a cyclone. The data were recorded by a data acquisition system (NI cDAQ-9172, National Instruments) and processed in the LabVIEW 8.6™ software. A calibrated peristaltic pump (Cole Parmer, Masterflex L/S) was used to deliver the liquid binder to the two-fluid spray nozzle (SU12A, Spraying Systems). Compressed air provided by a compressor (1.1 kW, Schulz) was used to perform the binder atomization.

2.3 Agglomeration experiments

The agglomeration experiments were carried out using the following operational conditions: 0.4 kg of raw RPC, air temperature at 65 ºC, pulsation frequency at 5 Hz and fluidizing airflow at 0.13 kg/min. Aqueous solution of hydrolyzed collagen (HC) at concentrations of 10 and 30 % (w/w) were used as binder. The solutions were fed at a flow rate of 1.5 mL/min and at a pressure air of 80 kPa. Firstly, the raw RPC was fluidized and heated during approximately 8 minutes. Subsequently, 120 mL of liquid binder was sprayed onto the particles. After that, the product was dried until the moisture content similar to the raw material. During the tests, the particle size was monitored in-line using a probe (Parsum IPP70, Chemnitz, Germany), which apply the spatial filter velocimetry principle to measure the ‘‘chord’’ (size) of the particles. Details of this methodology and the probe accessories were described by Silva and Taranto (2015). The particle size in volume obtained by the Inline Particle Probe 7.14 software were sent to LabVIEW™ 8.6 software via OPC Server protocol, procedure developed by Silva and Taranto (2015). The size distributions were also obtained by Parsum probe. The Parsum probe measures the size distribution between 50 to 6000 µm. The size of the ring buffer used in the Parsum measurements was set at 5000, i.e., 5000 particles are used in each measurement. The process yield (Yld) was calculated by the ratio between the sample mass remained in the RPFB (m_{final}) and the sample mass of raw material ($m_{initial}$), on dry basis. The mass fraction lost by fines entraining (m_e) and the mass fraction from lumps (m_l) or incrustation (m_{inc}) were discounted, according to Eq(1).

$$Yld(\%) = \frac{m_{final}}{m_{initial}} = \frac{m_{initial} - (m_e + m_l + m_{inc})}{m_{initial}} \times 100 \quad (1)$$

The evaporation efficiency (R) was calculated based on the relationship between the output absolute humidity (Y_{out}) and the absolute humidity measured in the adiabatic saturation (Y_{sat}), i.e., at the air wet bulb temperature, giving by $R = Y_{out}/Y_{sat}$ (Kage et al.,1996). Dewettinck et al. (1999) also defined evaporation efficiency (E) similarly, taking into account the humidity of the intlet air (Y_{in}) given by $E = Y_{out} - Y_{in}/Y_{sat} - Y_{in}$. The adiabatic saturation condition was calculated as described by Silva and Taranto (2015).

2.4 Characterization of raw and agglomerated RPC

The moisture content (MC) of the samples was determined using a halogen moisture analyzer (HR83, Mettler Toledo). Protein content (PC) in raw and agglomerated RPC was determined by the Kjeldahl method (AOAC 1997), the nitrogen conversion factor used was 5.95 (Juliano, 1994). The wetting time for raw and agglomerated RPC was measured as the time required for the complete wetting and immersion of 3 g of the sample when placed on water surface (60 mL at 27 °C), according to Hogekamp and Schubert (2003). The flowability of the raw and agglomerated RPC was analysed in terms of Carr index (CI), given by $CI(\%) = \left[\left(\rho_{tapped} - \rho_{bulk}\right)/\rho_{tapped}\right].100$, as described by Turchiuli et al. (2005). The classification of powder flowability

based on the CI (%) value is as follows: < 15 (very good); 15 - 20 (good); 20 - 35 (fair); 35 - 45 (bad) and > 45 (very bad). Statistical analysis of the results was made by Tukey's test, at a significance level of 0.05. The particle morphology of agglomerated RPC was evaluated by scanning electron microscopy (SEM) using a scanning electron microscope (LEICA Electron Microscopy Ltd., Cambridge, England) at magnifications of 100 x and 1000 x. Laser diffraction (Mastersizer 2000, Malvern Instruments) was also used to measure the particle size of the raw RPC.

3. Results

3.1 Experimental results and in-line particle size measurements

The raw material showed moisture content, bulk and tapped density of 4.12 ± 0.31 % (w.b), 0.486 ± 0.01 g/cm^3 and 0.650 ± 0.01 g/cm^3, respectively. The characteristic sizes $D10_v$, $D50_v$ and $D90_v$ measured by laser diffraction were 9.55 ± 0.16 µm, 54.2 ± 0.98 µm and 148.5 ± 9.68 µm, respectively. The $D50_v$ and $D90_v$ were measured in-line by Parsum probe. The results obtained were 94.9 ± 3.36 µm and 162.2 ± 6.40 µm, respectively. Both of them are related to the average result obtained from 85 measurements.The median particle size obtained by laser diffraction is fairly close to the Parsum probe measurement size distribution limit. Because of that, the value measured by probe stands far above the value measured using laser diffraction. With the particle size increase, these effects are reduced and better measures are obtained.

The $D90_v$ value measured by probe (162.2 ± 6.40 µm) is very close to the value obtained by laser diffraction (148.5 ± 9.68 µm). This difference is accepted, as Parsum probe analyzes a sample of 0.40 kg and laser diffraction uses a sample of 0.01 kg. So, this probe performed a much larger measurement over a ring buffer of 5000 particles, as discussed by Silva and Taranto (2015).

The evolution of the median particle size ($D50_v$) in volume is shown in Figure 1. Wetting of the particles can be observed during binder atomization. At the end of the agglomeration there was an increase in the moisture content from 4.12 ± 0.31 % (w.b) to 5.81 % (w.b) (Test 1) and 5.33 % (w.b) (Test 2). In general, the binder atomization together with lower drying temperature (65 ºC) makes the surface of the particles wet and stickier, thus providing the adhesion, consolidation and growth mechanisms. As a result, particles higher than those of raw material were produced. In addition, it can be noticed that the binder concentration presented a strong influence on the increase of the median particle diameter. When the binder concentration used changed from 10 to 30 %, the particle size increased from 240.7 ± 4.7 µm to 386.7 ± 7.9 µm, at the end of the agglomeration, as shown in Figures 1 (a) and (b). Dacanal and Menegalli (2010) observed the same when investigating the agglomeration of soy protein isolate in a pulsed fluid bed. The authors verified that an increase of binder concentration (maltodextrin) from 10 to 50 % promoted particle enlargement from 390 to 470 µm. Similar observations were also made by Jinapong et al. (2008) who reported that an increase of binder concentration (maltodextrin) from 0 to 10 % resulted in larger soymilk granules. The drying resulted in granules with moisture content similar to raw material, of 4.08 and 3.94 % (w.b), for tests 1 and 2, respectively.

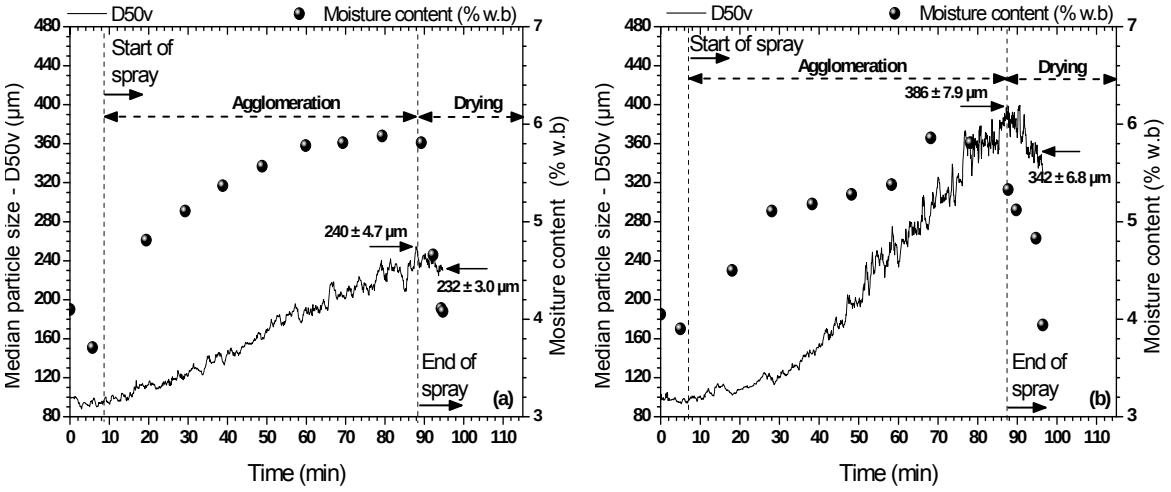

Figure 1: Evolution of median size ($D50_V$) in volume: (a) test 1 - C_{lb}: 10 % and (b) test 2 - C_{lb}: 30 %.

The particle growth rate was larger when higher binder concentration was used. Growth rate showed a linear behavior over the time at the test with the lower binder concentration, Figure 1 (a). However, two phases were

verified when higher binder concentration was used, Figure 1 (b). The growth rate seems to be parabolic in the first twenty minutes of agglomeration. It should be related with the wetting and nucleation step of the agglomeration process, which may have been more pronounced in this condition. This step is when the liquid binder and powder surface first come into contact and form the initial nuclei (Iveson et al., 2001). After that, i.e., from 120 µm in the median particle size, a quick increase of the particle growth rate could be observed and the growth rate became linear over the time.

In the drying phase, both curves showed a downward trend, indicating the reduction of the granule size, due to its breakage. Moreover, the reduction of the granule size was more pronounced at higher binder concentration, which is probably related to the easy break up of large particles. At the end of the process, the median diameter of agglomerated products was 328 and 530 % larger than the initial size of raw material, for tests 1 and 2, respectively.

The results of size distributions using comparisons with Tyler sieves obtained by Parsum probe are shown in Figure 2. In Figure 2 (a) a higher percentage of fine particles (< 75 µm) could be noticed before the binder atomization. The median particle size was between the sieves 75 and 150 µm. However, this value stands above the real size, since the raw RPC has a greater amount of fine particles (< 50 µm), with a fairly close size to the measurement limit, as previously discussed. At the end of the process, the fractions of coarse particles increased, as shown in Figures 2 (b) and (c). Figure 2 (b) illustrates the size distribution for binder concentration at 10 %. It could be seen a reduction of the fine fractions (<150 µm) as well as an increase of the coarse particles greater than 250 µm. The median particle size was between the sieves 150 and 250 µm. When the binder concentration was increased to 30 %, Figure 2 (c), the median size was between the sieves 350 and 420 µm, indicating stronger agglomeration. In this condition, it was also verified the presence of coarse fractions (> 420 µm), producing a broader distribution and a fraction with smaller particles between the sieves 150 and 250 µm suggests a bimodal size distribution.

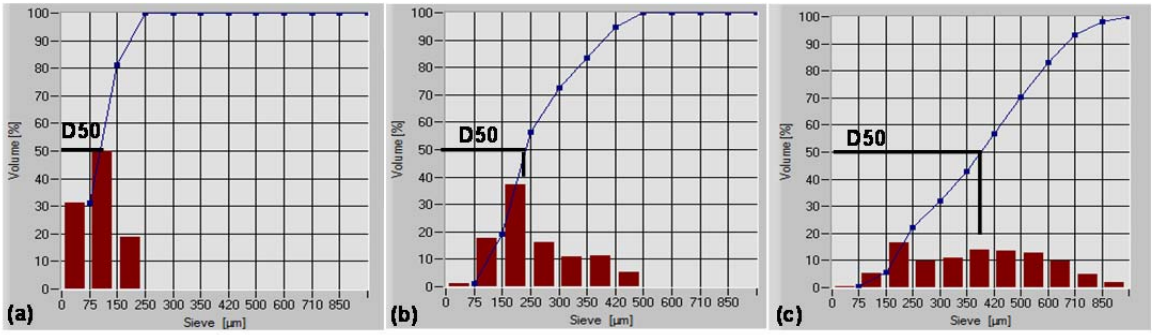

Figure 2: Particle size distributions at fractions (histogram) and the cumulative curve obtained by Parsum probe: (a) beginning (raw material), (b) end of test 1 and (c) end of test 2.

Figure 3 shows the evaporation efficiency parameters (R and E). These parameters are effective to indicate an unstable region due to the excess of moisture in the bed, represented by values closely to the unit. Figures 3 (a) and (b) shows that the values of R and E presented the same tendency in both conditions studied.

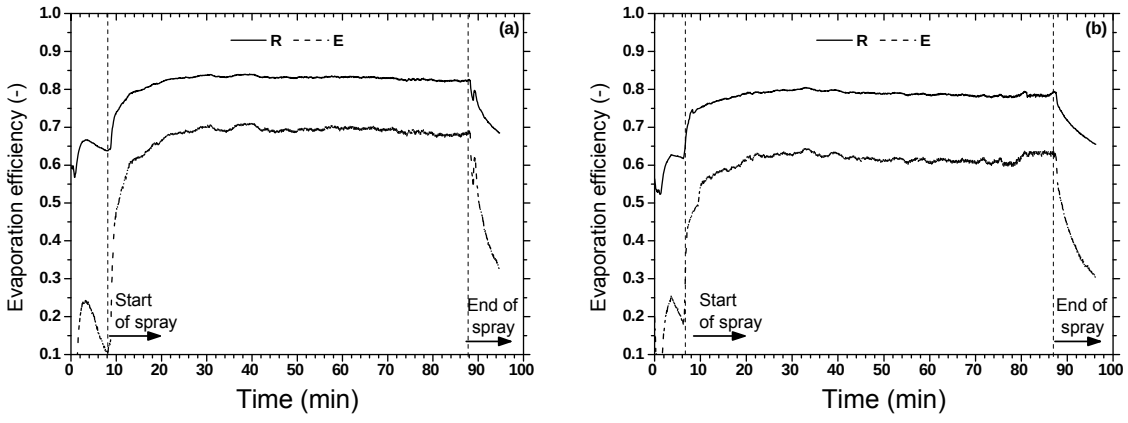

Figure 3: Evaporation efficiency during the process: (a) test 1 - C_{lb}: 10 % and (b) test 2 - C_{lb}: 30 %.

The values of efficiency increased when the atomization started and achieved constant levels after 10 minutes of atomization. At the same time, the growth of particles begins to occur, as confirmed by size analysis shown in Figure 1. The maintenance of energy balance between the amounts of heat introduced into the bed with the moisture evaporation rate presented a strong tendency to particle agglomeration. Nonetheless, higher values of efficiency suggest that the input air temperature (65 °C) is not sufficient to provide the entire drying of the particles, so that the particles are wet enough to agglomerate. The efficiency values reduced drastically with the beginning of the drying, showing an improvement in drying capacity of the bed.

The process yield obtained for the test 1 (C_{lb}: 10 %) and test 2 (C_{lb}: 30 %) was 45.1 and 55.5 %, respectively. These results revealed that higher process yield is obtained when higher binder concentration is used. The product losses occur mainly due to fines entrainment. The atomizing pressure and the pressure used to operate the Parsum probe also contributed to the entrainment of the fine particles. A higher binder concentration aids the formation of larger granules, promoting lower rates of fine particles entrainment. Dacanal and Menegalli (2010) observed the same during the agglomeration of soy protein isolate. The authors reported that the granules produced from drops with higher binder concentration dry faster, and thus improve the particle growth rate. In opposite, the growth rate decreases when drops with lower binder concentration are used, resulting in a process with a higher entrainment of fines and a lower process yield.

3.2 Characterization of raw and agglomerated RPC

Table 1 compares the chemical and physical properties of raw and agglomerated RPC. The protein content (PC) of the agglomerated products was higher than those of the raw RPC as expected, because the binder is a hydrolyzed protein. No significant difference was observed in the PC when different binder concentrations were used. The CI value for the raw RPC was 25.3 ± 1.2 %, while for the agglomerated RPC this value was 15.7 ± 0.6 % (test 1) and 17.0 ± 1.0 % (test 2), which evidences that the flowability level changed from fair to good. The increase in binder concentration did not influence the flowability of the agglomerated RPC, since no significant difference was verified in the CI values. The agglomerated product showed shorter wetting time than the raw RPC. The complete wetting was achieved in less than 17 s (test 1) and 35 s (test 2), which evidences its greater capacity to absorb moisture. In contrast, the raw RPC took 140 s to reach complete wetting. The increase in the particle size allowed the particles to fall and disperse easier, which was observed visually. Further, it was verified that higher binder concentration also resulted in higher wetting time (34 s). Probably, this is because the binder is not completely immersed into water, as reported by Andreola et al. (2015). The authors reported that when the hydrolyzed collagen was spread on the water surface a large fraction of the material remained on the surface of the liquid, forming a thick layer, which made it difficult the dispersion of the particles. So, higher concentration of this binder resulted in a product with longer wetting time. The improvement of the powder flowability and reduced wetting time was described in others agglomeration studies (Dacanal and Menegalli, 2010; Toumi et al. 2013).

Table 1: Chemical and physical properties of raw and agglomerated rice protein concentrated

Test	PC (% d.b)	WT (s)	CI (%)	Flowability
Raw RPC	72.92 ± 1.01^{b}	140 ± 2.8^{a}	25.3 ± 1.2^{a}	Fair
1 (C_{lb}: 10%)	82.33 ± 0.86^{a}	17 ± 1.4^{b}	15.7 ± 0.6^{b}	Good
2 (C_{lb}: 30%)	80.65 ± 1.30^{a}	35 ± 1.4^{c}	17.0 ± 1.0^{b}	Good

C_{lb}: liquid binder concentration (% w/w); PC: protein content (% d.b.); WT: wetting time (s); CI: Carr index (%). Mean values in the same column with different letters are significantly different ($p < 0.05$).

Figure 4 shows the SEM images for the agglomerated RPC at operating conditions studied. Agglomerated RPC showed an irregular structure and presented wrinkled and porous surface. It can be seen that higher binder concentration (30 % w/w), Figure 4 (b) and (d) resulted in larger granules than those formed using lower binder concentration (10 % w/w), Figure 4 (a) and (c).

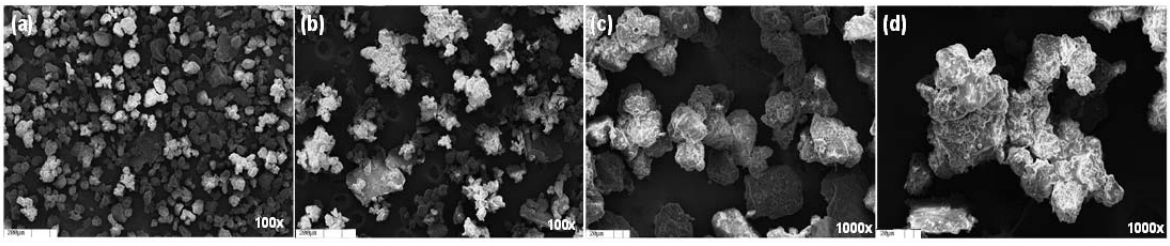

Figure 4: SEM micrographs: (a) and (c) test 1; (b) and (d) test 2.

4. Conclusions

The use of a rotating pulsed fluidized bed was feasible to perform the rice protein concentrate agglomeration under operating conditions studied, resulting in larger granules with low moisture content. HC solutions at concentrations studied could be applied as binder to agglomerate RPC, allowing the non-use of adjuvants substances like carbohydrates in the production of a powder protein. Agglomeration of RPC produced larger particles with high flowability. The size enlargement also resulted in an improvement of instant properties that was characterized by the higher wettability of the agglomerated product. Drying efficiency values closer to the unit showed a greater tendency of agglomeration. In line size monitoring allowed a better understanding of the particle growth during the agglomeration. The monitoring of the particle size evolution is useful, since it is possible to set the stopping point of the process based in the particle size desired. Overall, the agglomeration process is a useful method to produce instant RPC, which can be used as a potential ingredient in foods or pharmaceutical formulations, especially in hypoallergenic formulations.

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