Spinning Disc Reactor to produce Nanoparticles: Applications and Best Operating Variables

Angelo Chianese[a,*], Alfredo Picano[a], Marco Stoller[b]

[a]Labor srl, Via G. Peroni 386, 00131 Rome, Italy
[b]Sapienza University of Rome, Via Eudossiana 18, 00184 Rome, Italy
 angelo.cianese@fondazione.uniroma1.it

A spinning disc reactor (SDR) is a useful equipment to produce monodisperse nanoparticles with controllable properties, as particle size and particle size distribution. Since the late 90s, this technology has been successfully proven for the reaction and solvent-antisolvent precipitation process. This paper reviews the works on the use of SDR to produce inorganic and organic compounds. Firstly, the more significant works on the subject are presented concerning the produced compound, then the factors influencing the process performances are examined in the light of the results in the literature. Finally, some considerations on the fluid stream's hydrodynamics modelling along the disc surface are attempted.

1. Introduction

This paper concerns the use of the spinning disc reactor technology for producing nanoparticles by precipitation-reaction. The liquid reagents streams are directed towards the centre of the disc, which is rotated rapidly (300 and 3000 rpm) resulting in a thin fluid film (1 to 200 nm). The thickness of the fluid layer and the large contact area between it and the disc surface induces high heat and mass transfer. Finally, the drag forces between the moving fluid and the disc surface enable very efficient and rapid mixing (micromixing) among the reagents in the liquid streams flowing along the disc surface. The production of nanoparticles, defined as particles less than 100 nm, implies applying a procedure that fosters the nucleation rate and limits the growth and agglomeration rates. When the limiting reagent achieves its maximum value, supersaturation and reaction rate reaches the ultimate value, and the nucleation rate is maximized. A prerequisite is to attain a micromixing among the reagents, which is the complete mixing of the inlet fluid streams into the reactor. Typically, the micromixing time is lower than 1 ms.

The spinning disc reactor (SDR) is the more flexible and straightforward equipment for the mixing intensification among liquid streams flowing over its surface. It may be operated in continuous mode to produce a commercial amount of the solid product. Moreover, it is inherently safer than the traditional stirred tank reactors and its operation at lab scale can be easily scaled up (Bodhoo, 2013). An SDR 500 mm in diameter can typically process about 150 kg/h of material with water-like viscosity. Fig. 1 shows an SDR produced by LABOR srl.

The fluid streams are fed over the disc at the central point or along the disc radius. The feeding point criterium is a compromise between the maximum local mixing and the residence time to be attained for the best solid production process performances. The disc may be maintained at a suitable temperature by a heating coil put under the disc or circulation of a thermostatic fluid stream within the disc plate. The rotation speed is usually variable and strictly controlled. The disc, usually made of copper or stainless steel, has a smooth or grooved surface. In the latter case waves and ripples are generated within the disc's liquid layer to increase the turbulence. In some works, a vertical SDR with a horizontal rotating shaft is adopted to allow an easier discharge of the slurry. A novel and higher dissipation energy equipment consist of two coaxial rotating discs (DSDR) (Farahani et al., 2017). The upper disc may rotate both clockwise and counter-clockwise. The Authors investigated the performance of DDSR in the synthesis of barium sulfate through the precipitation process. Smaller particles with narrower particle size distribution are some advantages of this new equipment.
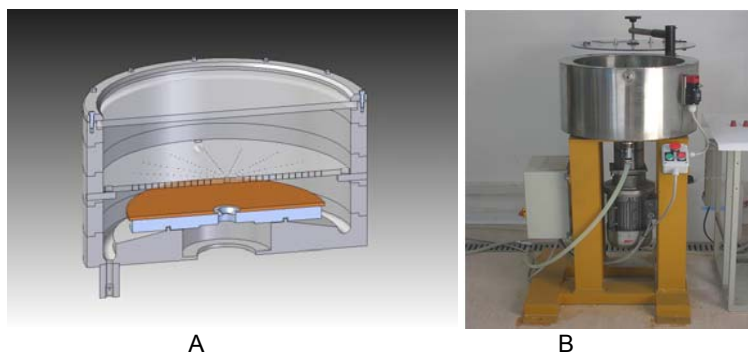
Figure 1. SDR designed and constructed by LABOR srl, (A) scheme of the disc and the plate for the location of the feed stream; (B) photo of the SDR pilot plant

2. Nanoparticles produced by SDR technique

The spinning disc reactor (SDR) technology, also named spinning disc processing (SDP), has been introduced at the University of Newcastle by the pioneering work of C. Ramshaw on process intensification (PI), on the improvement of heat transfer on rotating surfaces (Jachuck and Ramshow, 1994). They successfully experimented its use for a pre-polymerization for the manufacture of polystyrene (Jachuck and Ramshow, 1997).

At the end of the last century, from the collaboration between the University of Newcastle and the Sapienza University of Rome, the SDR technology was applied to barium sulfate reaction-precipitation (Cafiero et al., 1999). Even in the absence of more advanced investigation instruments, now available, as nano-sizer it was evident the SDR's advantage to produce nanoparticles by precipitation-reaction. During the last 20 years, the SDR technology has been applied to produce various nanoparticles through reaction-precipitation or by the solvent-antisolvent (S-AS) process. Both inorganic and organic products were produced, like composite nanoparticles for the drug release, battery application, etc. In Table 1, a list of material produced as nanoparticles by the SDR technique is shown.

Table1. Nanoparticles produced by SDR technique and the adopted operating parameters

Material	Authors	Disc diameter cm	No. Feed points/distance from centre, cm	Finest particle nm	RPM at finest particle
Ag, silver	Kisyelovab et al. 2016	9	1/2	28 nm	900 rpm
Ag	Tai et al. 2009	19.5 Vertical disc	1	< 10 nm PVP	> 500 rpm
AgCl, silver Chloride	Dabir et al. 2014	15 - 20	1	37 nm	1500 rpm
AgI, silver Iodide	Liu et al. 2012	19,5	1	66 nm PVP	4000 rpm
BaSO4, barium sulfate	Cafiero et al. 2002	50	1	700 nm	1000 rpm
BaSO4	Farahani et al. 2017	20	4/1,4,7,10	23 nm	4750 rpm
Ca10(PO4)6(OH)2, Hydroxyapatite	De Capraris et al. 2012	8.5	1/2,3	80 nm	1400 rpm
Cu, copper	Marchetti et al. 2019	8.5	2,4/2.5	26 nm	1400 rpm
Cu	Ahoba-Sam et al. 2018	10	1	3.2 nm	2400 rpm
CuO, copper oxide after calcination	Chang et al. 2011	20 Vertical disc	1	45 nm	4000 rpm
Fe nZVI, iron nanozerovalent	Vilardi et al. 2019	8.5	5/1.5, 2, 2.5, 3, 3.5	28 nm	1400 rpm
Fe3O4, magnetite	Chin et al.	20	1	10-200 nm	2000 rpm

encapsulated in chitosan	2006				
Fe3O4, magnetite	Chin et al. 2008	10	1	3-5 nm	2500 rpm
FeO4	Haseidi et al. 2016	10	1	6 nm	5000 rpm
KNO3, potassium nitrate	Vilardi et al. 2017	8.5	1 with recirculation	< 250 nm	140 rpm
Mg(OH)2, magnesium hydroxide	Tai et al. 2017	12	1	47 nm	2000 rpm
Pd, palladium	Zou et al. 2011	10	1	160 nm	1500 rpm
S, sulfur core to be coated with TiO2	Dell'Era et al. 2019	8.5	1	60 nm	1400 rpm
TiO2, titanium dioxide	Mohammadi et al. 2014	30	3/0,5,10	< 5 nm	1200 rpm
ZnO, zinc oxide	Stoller et al. 2020	8.5	4/1.5, 2, 2.5, 3	50 nm	1400 rpm
ZnO	Hartleb et al. 2007	10	1	1 nm	3000 rpm
β-carotene	Anantachoke et al. 2006	10	1	40 nm	1000 rpm
Chitosan for drug loading	Loh et al. 2010	10	1	20 nm	1000 rpm
Curcumin	Khan et al. 2014	15,18	1 S-AS process	220 nm	1500 rpm
Nimesulide	Rathod et al. 2018	18	1 S-AS process	192 nm	300 rpm
Starch	Cana et al. 2019	30	1 S-AS process	90 nm	1200 rpm

Table 1 shows in order, metallic, inorganic compounds, and organic ones. Some nanoparticles, after production are functionalized by coating with chitosan (Chin et al., 2006), oleic acid (Haseidl et al., 2016), or titania (Dell'Era et al., 2019) to their more specific use. Looking at the characteristics of the adopted SDR, we notice that the disc diameter is in the range of 8.5 – 30 cm, apart from a value of 50 cm used to produce barium sulfate in the first application (Cafiero et al., 2002). The applied rotational velocity of the disc is between 500 and 5000 rpm. The relatively small disc diameter adopted is due to these main reasons: the need to limit the residence time of the born nanoparticles over the disc and thus of their size growth, and to achieve a high disc rotation per minute, suitable to attain the micromixing of the fluid streams over its surface. In some cases, there is a gas atmosphere inside the spinning disc case to accomplish the reaction. Examples are the reduced atmosphere of H_2 in Pd's production (Zou et al., 2011) and that one of carbon dioxide for carbonation of calcium hydroxide to produce calcium carbonate (Tai et al., 2005).

The SDR technique's performance in terms of average nanoparticle size is very satisfactory: for the inorganic compounds, besides a few cases, the value is lower than 100 nm and often smaller than 10 nm. Nanoparticles of organic compounds produced by the S-AS process exhibit higher size values, mainly because a harder supersaturation control. The comparison between the performances of a stirred tank reactor (STR) and an SDR shows that the better fluid mixing of the SDR leads to a substantial size reduction of the produced solid particles, but to an increase of the energy consumption for unit weight of the solid product because of the higher induced turbulence. Cafiero et. al. (2002) reported a value of 115 W/kg for the precipitation-reaction of $BaSO_4$, whereas Khan et al. has evaluated an energy consumption of 473 W/kg of curcumin produced by an S-AS process (Khan and Radhot, 2018).

3. Main control parameters for SDR

The main SDR control parameters are the disc speed, the nature of the disc surface (smooth or grooved), and the feed liquid streams' flow rates. Further parameters are the attained supersaturation of the product that precipitates, the number of the feeding point and their location, finally the adopted, if any, stabilizing agent. Obviously, supersaturation has a direct effect on the nucleation rate of the solid. Its increase due to higher concentrations of the reagents or lower ratio S-AS values leads to reducing the particle size (PS). The drawback is the increase of the agglomeration rate, which the presence of dispersion agents should face, often adopted.

Disc rotation speed increases both micromixing and the process yield, whereas it reduces the liquid residence time. Ahoba-Sam at al. (2018) has shown that increasing the rotation speed and thus shortening the residence time leads to both a reduction of growth and agglomeration of nanoparticles, thus reducing their size, since the fluid pattern is like a plug flow. In the production of TiO_2, a rotation speed increase from 200 to 1200 rpm determined a micromixing time reduction from 3.5–6 ms, as a function of the liquid flow rate, down to 0.3 – 0.5 ms, correspondingly the median PS decreases from 16 to 5 nm (Mohammadi et al., 2014). Almost a linear PS decrease for nimesulide in the range 500-3000 rpm was observed by Rathod et al. (2018). The effect of the rotation speed is not evident for some processes. PS slightly decreased for $Mg(OH)_2$ from 58 to 48 nm for an increase of rotational speed in the range 400-2000 rpm (Tai et al., 2007) and it was not significant in the precipitation of silver in the range 500-4000 rpm (Dabir et al., 2015). For magnetite, a continuous decrease from 10 nm to 6 nm occurred in the range 500-5000 (Haseidl et al., 2016). Still, the drawback was the reduced magnetic response, similar behaviour has been observed by Chin et al. (2008), but the reduced magnetizability was in this case almost negligible.

The effect of the disc surface, i.e. smooth or grooved, has been examined by several Authors with contradictory results. Smooth and grooved discs are equivalent in the production of starch (Sana et al., 2019), there is a benefit in terms of PS reduction for grooved disc 15 cm in diameter, but not for the disc 18 cm in diameter (Rathod et al., 2018). The PS of ZnO is lower with the smooth disc (Hartlieb et al., 2007). In conclusion, the disc surface choice in the absence of definite indications must be left to the experimentation.

A factor initially neglected in the experimental works and later always more often investigated has been the feed location. There are two aspects to be considered the location point over the disc, i.e. the distance from the disc centre, and the splitting of each fluid stream. The fluid stream moving away from the disc centre is fed where high turbulence takes place, whereas the fluid stream splitting reduces the local concentration of the generated nuclei and the agglomeration of the particles as well (Marchetti and Stoller, 2019). Both aspects appear to be advantageous to reduce the produced nanoparticle size. The effect of the feed distribution pattern was explored by Farhani et al. (2017) by introducing the feed solution up to 16 points along the disc periphery by a slight decrease of the PS. By moving the location feed point from 2 to 3 cm for a disc 4.25 in radius, a significant reduction of PS was observed by de Capraris et al. (2012). Vilardi et al. (2019) observed a PS reduction by putting the feeding point away from the centre up to 3 cm, a subsequent displacement to 3.5 cm was not advantageous.

The importance of the use of a stabilizing agent was shown by many Authors and affected many process performances, among them the stabilization of the solid solution, the PS reduction, and the increase of the process yield and the crystal form (Liu et al., 2012). It is interesting to point out that the use of NaHMP in the production of CuO nanofluids (NF) improved both solution stabilization and NF conductivity (Chang et al., 2011).

Moreover, a surfactant can be an important parameter in the "bottom-up" synthesis of nutraceutical nanoparticles (Annatachoke et al., 2006).

Some Authors have proposed some modification in the geometry of the configuration of the SDR. Tai et al. (2007) have adopted a disc with a horizontal axis to an easier discharge of the solid suspension. Farahani et al. (2017) carried out the precipitation-reaction of barium sulfate using a double coaxial disc 20 cm in diameter. The upper disc can be drive counter-clockwise, whereas the lower disc may round clockwise and counter-clockwise. The location of the feeding point was also examined, and the minimum PS of 23.4 nm was obtained by operating at 4750 rpm with a feed location point 7 cm away from the disc centre.

4. Model of hydrodynamics of the liquid streams on the SD surface

Identifying the local turbulence, i.e. the local micromixing times and the residence time of the fluid all over the disc, can be usefully done using the liquid film's modelling hydrodynamics over the disc surface.

The first attempt to develop such a model in 2D has been made by Bathelia et al. (2009). More recently, the Sapienza University of Rome's research work team developed a hydrodynamic model for the precipitation-reaction of hydroxyapatite (HPA), a smooth disc 8.5 cm in diameter was adopted (de Caprariis et al., 2012). The film thickness varies from 300 nm near the feed injection to 50 nm at the disc extremity. The feeding points of the two reagent fluid streams are located 2 cm from the disc centre. The disc diameter is about three orders of magnitude greater than the maximum film thickness; therefore, a 2D model was developed using a fine grid on the vertical direction and a large one in the radial direction. The hydroxyapatite concentration profiles in the liquid phase and the local reaction rates, at a film height of 35 μm, are shown in Fig. 2 (de Caprariis et al., 2015). The obtained results seem realistic and clearly show that the maximum reaction rate corresponds to the feeding points, whereas the produced HPA reaches the maximum concentration at the disc periphery.
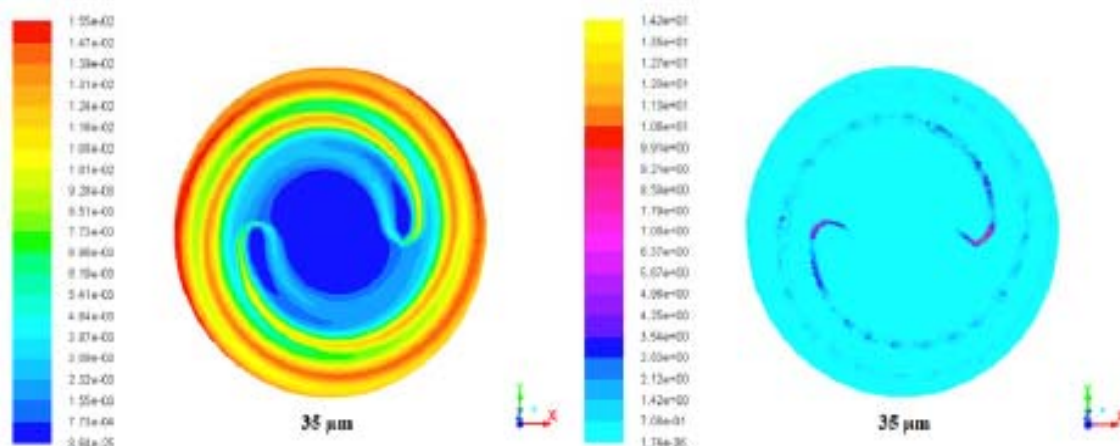
Figure 2. Fluid dynamics over the SDR surface in the production of hydroxyapatite (HPA)at a film height of the liquid layer of 35 μm: (A) HPA concentration; (B) reaction rate

5. Conclusions

Twenty years of research has shown the great utility of SDR technology in producing organic and inorganic nanoparticles. The main success reasons are excellent mixing between liquid streams, which enables local micromixing, effective heat and mass transfer, ideal for gas-liquid reactions, the potential for sequential reagents for tailored products, and continuous operation mode. After the traditional use for the precipitation-reaction process, more recent applications have concerned solvent-antisolvent precipitation. The energy required by this technology to allow micromixing conditions is higher than that one needed by the STR equipment. Still, any comparison between the two technologies in terms of the reduced size of the produced particles is in favour of SDR. Finally, modelling of the mixing process has been successfully studied in two dimensions. New advances are expected in the future for a complete simulation of the precipitation process, including the birth, growth, and agglomeration of the solid particles.

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