The Feasibility Analysis To Produce Toner Using CO_2 Supercritical Fluid As Gas Anti-Solvent

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Abstract

A novel method of producing toner is proposed in the article. The solvent which macromolecule resin for producing toner can be dissolved in is regarded as the processing medium at special temperature and pressure, the pigment and CCA （Charge Control Agent）etc. used for producing toner can be dispersed or dissolved partly in the mucous resin solution, then through putting CO_2 supercritical fluid in and controlling system parameter, the toner particles would be formed as while as the solvent is extracted by the CO_2 supercritical fluid. Also the method which materials choosing, system designing and reusing of the processing medium is proposed in the article.

1 Background

We have presented the RESS (Rapid Expansion of Supercritical Solution) method to produce toner in ICISH'04. Till now, we have known that Sharp company (Japan) & Huinon Toner company (China) had applied the patents, so the new technology which using the CO_2 supercritical fluid (SCF-CO_2) as the processing medium to produce toner has been attracted attention by scientists and enterprisers. For the technology, the best advantage is that the processing medium & the waste produced in manufacture can be recycled, and benefit for saving energy and protecting the environment.

But some problems would be met when the RESS is used for manufacture toner, the macromolecule resin[1] which include –OH & –COOH etc strong polar group or –OH & –COOH groups attached directly to the benzene ring can be dissolved in SCF-CO_2, but the pressure normally should be over 40MPa. In this way, the equipment invest is expensive, the product cost would be high. Even the excellent toner can be produced by RESS method, it's difficult to popularize the technology.

So in this article, we present the GAS (Gas Anti-Solvent) method for producing toner.

Using GAS method to manufacture toner, first the macromolecule resin used for toner should be dissolved in some organic solvent, and the pigment, CCA etc. materials used for toner must be dispersed uniformly in the solution containing resin, then the SCF-CO_2 is mixed with toner solution. As the organic solvent can be dissolved in SCF-CO_2 at normal temperature and lower pressure, but the resin, pigment, CCA etc. materials can't be dissolved in SCF-CO_2, so the solute toner will be separated out and formed to be toner particles. The toner particles produced by this method is global, it's shape is similar as the toner particles produced by chemosynthesis method. The organic solvent and the SCF-CO_2 can be separated by separator, so they can be reused.

2 The Principle Of Prescription Designing & Material Choosing

2.1 Transplanting Conventional Prescription

It has mature experience now that toner is produced by mechanical mulling method, and the prescription has been the popular knowledge in the industry. So the prescription of conventional manufacture method should be used in first when SCF-CO_2 GAS method is used to produce toner. But it should be paid attention that the quantity of micro-powder F in the prescription would be adjusted according to the actual proportion.

2.2 Choosing Resin

We should consider processing manufacturability of the resin, such as Tg, Ts, Tm, molecular weight & it's distribution etc. when toner is manufactured by conventional mulling method. The styrene acrylate polymer is regarded as the main material used for toner because it is friable. But if toner is manufactured by SCF-CO_2 GAS method, it don't need mulling, more kinds of resin can be chosen, for example, polythene can be the first research object because the cost is lower and it is more healthy for human.

2.3 The Requirement of Pigment And CCA

As the pigment, CCA used for toner must be dispersed uniformly in the organic solution containing resin, the pigment, CCA particles should be easy to be dispersed in organic solution, the agglomerate particles can be rapidly dispersed while being mixed round and have a nice affinity for resin. If the pigment is magnetic iron oxide, the surface of iron oxide should be processed first. The pigment particles size must can't be larger than 0.3μm and the CCA particles size must can't be larger than 1.5μm.

2.4 Choosing Solvent

One of the key for GAS method is the solvent which resin can be dissolved in at normal temperature. From previous experience, toluene, xylene, styrene or clove oil can be used as solvent to dissolve toner produced by styrene acrylate polymer.

As a GAS research example, DixonD.J[2] used toluene as solvent to dissolve polystyrene, he used SCF-CO_2 as GAS, round polystyrene particles with minisize holes were made. Therefore toluene is worth to research as solvent to dissolve styrene acrylate polymer.

3 The Designing & Processing Of The Experiment System

3.1 System Structure

For protecting entironment, no discharge & recycle, the figure of GAS method for producing toner is as follow:

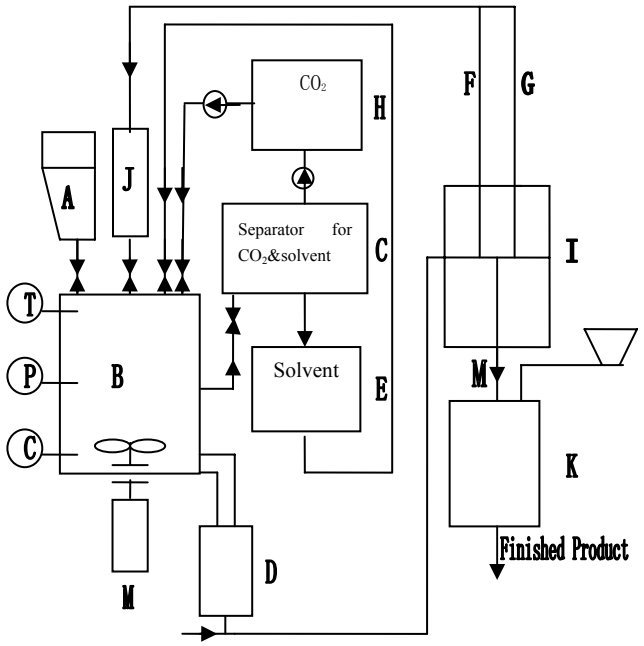

Figure 1 Cycle theory of GAS method for producing toner

A: material hoper, B: mixing kettle, C: Separator for CO_2 & solvent, D: Collector for toner particles, E: Collector for solvent, H: CO_2 storage, I: Classifier (F is micro-powder, G is large particle powder, M is toner powder which D50=7～8μm), K: Toner surface processor, J: Powder collector

B, H are equipment able to bear high pressure (20MPa) in the system, their keys are choosing force pump and airproof technology. Magnetic force driver is drived by electromotor while mixing in the mixing kettle, SCF-CO_2 is prevented to be out. I is inertial classifier in the system, the toner particles whose size are less than 5μm or larger than 9μm would be separated out.

3.2 Processing

i) Resin, pigment, CCA etc. are confirmed according to the above principle. The raw mixture is added to B from A, and the collected unregular powder particles which is too tiny or too large last time are also added into B, the material will be mixed uniformly by the blender.

ii) Put the solvent into B according to presetting concentration, start the blender and the resin can be dissolved well and the pigment, CCA can be dispersed well.

iii) CO_2 is added slowly to B from H. After the blender starts, the pressure in B is up to presetting range, the blending speed is up to presetting value, the blender stops till that the toner solute is separated out fully.

iv) As the pressure in B and C are difference, the mixture of CO_2 and solvent would go to C as soon as the valve is opened, then the CO_2 and the solvent would be separated naturally. The CO_2 can be poured into H by high-pressure pump, the solvent can pour into E, all of them can be reused.

v) After the mixture of CO_2 and solvent are all out of B, Start the blender, let the toner powder pour into D, then they would be sent to classifier, the large particles G & tiny particles F are back to J and prepare to be redissolved next time. The regular toner particles M would be sent to K, their surface are processed by nanoscale SiO_2, we will get the finished product – toner.

4 Analysis Of Conditions Affecting Toner Particle Size

4.1 Solution Concentration

From Dixon's experiment, the polymer solution concentration is closely related to the toner particle size. The fundamental experiments should be done under 50℃ and the CO_2 pressure should be less than 7.3MPa, series graphs of toner particle size and solution concentration would be gotten.

From Dixon's experience, the particle size would be larger with the increase of solution concentration, so to adjust the solution concentration is one of the way to control the particle size.

And Dixon and others has developed a new method PCA (precipitation with compressed fluid antisolvent) also, using the technology, polymer can be manufactured into round particles with minisize holes or hollow spherical particles, the hollow size is related to the solution concentration, the hollow size would be larger with the increase of the solution concentration. If the technology can be transplanted to toner producing, it would be profitable for the toner particles surface processing by nanoscale material, and also helpful for increasing the transfer and expending less on toner.

4.2 The Pressure Of CO_2 and The Blending Speed

Before the CO_2 being sent to the mixing kettle, blending the solution containing toluene under 50℃, the resin can be accelerated to dissolve in the solution and the pigment, CCA can be dispersed best.

The important subject for research is the way CO_2 added to the solution containing resin. From 北村[3]'s experiment that sulfathiazole ($C_9H_9N_3O_2S_2$) can be separated out to particles from solution containing alcohol when the CO_2 is as GAS, the particle size is larger with the increase of CO_2 pressure. We suggest to increase the pressure gradually as while as starting to blend.

For preventing too much particles less than 5μm formed under lower CO_2 pressure, the blending speed should be much low. And increase the speed gradually with the increase of CO_2 pressure, the conditions about pressure and speed when toner particles size is about 7μm would be discovered after time after time experiments.

5 Summarize

From Dixon DJ and 北村's research experience about using GAS method to produce polymer and other organic compound particles, our suggestion about using GAS method to produce toner is feasible. The key is discovering the relation of particle size and blending speed at room temperature (20℃-35℃). The conditions about solution concentration, CO_2 pressure in mixing kettle and blending speed & time when toner particles size is about 7μm would be discovered on the base of above experiential data. Although we have no finally result at present, but the research direction is certain that the material, processing medium can be reused and no discharge.

References

[1] Jingcheng Zhang, Supercritical Fluid Extraction, 2000，6.5:134
[2] DixonDJ,Luna-BarcenasG,Johnstonkp.Polymer, 1994, 35(18):3998
[3] 北村光孝 and others，化学工学シンポジウァシリ-ズ，1995，49：200