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Study of the performance of a membrane-based vacuum drying process

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ABSTRACT

The possibility of applying vacuum membrane distillation for drying polystyrene microparticles was recently confirmed and the best membrane unit design, named Vacuum Membrane Dryer (VMDr), identified. However, some limits in the production efficiency were underlined. Therefore, in the present work, further investigations to improve the performance of the previously developed and selected unit were carried out. In particular, the effect of mixing and air bubbling was analyzed. Moreover, the efficiency of the VMDr as function of the particles size, the membrane area and the membrane properties was studied.

1. Introduction

The drying of solid microparticles can be carried out in different devices, like press filters coupled to ovens, spray dryers, fluidized beds and vacuum dryers. Press filters suffer from the loss and deformation of particles, whereas both spray dryers and fluidized beds, in addition to the mechanical stress exerted on the particles, show limits in terms of particle size to be processed and need cyclones/bag filters to separate particles from the hot air sent for drying. Neither the press filters the spray dryers nor the fluidized beds are able to recover clean water. Conversely, vacuum drying is used in several industries (chemical, pharmaceutical, food, plastic, etc.) to remove and recover water (or other solvents) from moist materials, while obtaining a dried product. By working under vacuum, it is possible to promote the water evaporation already at low temperatures, so that also products sensitive to high temperatures can be efficiently treated. The vacuum pump is connected to the chamber where the material to be dried is loaded and the evaporated stream is condensed in a condenser located immediately before the vacuum pump. To work at the desired temperature, the material to be dried is usually in contact with a heated surface and the heat is transferred to the material by conduction.

There are different types of vacuum dryers that can be employed, depending on the properties of the feed to be treated and on the desired final product [1]. In tumble vacuum dryers, the material is loaded into rotating vessels that promote the mixing of the feed and its contact with the heated walls. A delumping bar can be operated intermittently to break up undesired agglomerates. Agitated vacuum dryers are based on stationary heated vessels, in which the feed is located, equipped with mixers. In vacuum belt dryers, the wet material is put onto a heated belt that slowly moves toward the product collector, where the dried material is discharged, after having passed through a crasher. Tray vacuum dryers consist of heated shelves and usually do not include units for mixing the feed nor breaking the agglomerates. Therefore, if needed, the agglomerates of dried material are broken outside the unit.

Regardless the specific type of vacuum dryer, the vacuum is applied at the feed chamber and there is a risk of product loss by vapor entrainment, especially when fine particles are treated. For this reason, vacuum dryers are always equipped with dust filters that usually have a nominal mesh size ranging from 1 to 5 μm. In last years, a lot of research has been addressed to the application of membrane contactors in different fields of industrial interest, like gas–liquid operations (e.g., CO_2 absorption), liquid–liquid extractions (e.g., metals removal from aqueous streams), distillation (e.g. wastewater purification; desalination) [2]. In membrane contactors, microporous hydrophobic or hydrophilic membranes are used to promote the mass transfer between phases, preventing their mixing. Each membrane micropore acts, in fact, as point of contact between the involved phases, providing a high interfacial area in a small volume (high compactness). With respect to traditional units, there is the possibility of varying independently the stream flowrates and the problems of flooding and foaming are avoided.

Membrane distillation belongs to the "membrane contactors family" and is based on the use of microporous hydrophobic membranes. The aqueous feed to be distilled is in contact with one side of the membrane and, due to the hydrophobic character, cannot

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penetrate inside its pores, that are liquid-free. At the other side of the membrane, depending on the specific configuration, a colder aqueous stream (Direct Contact Membrane Distillation – DCMD) or a sweep gas (Sweep Gas Membrane Membrane Distillation – SGMD) can be sent, in order to create a difference of water vapor pressure across the membrane and, then, to promote the water vapor transfer. Alternatively, an air gap can be created (Air Gap Membrane Distillation – AGMD) through which the water vapor migrates before being condensed on a colder surface, or vacuum can be applied (Vacuum Membrane Distillation – VMD). Membrane distillation, in its different configurations, has been widely studied for the treatment and concentration of water streams, included sea and brackish waters [3–16].

Recently, the possibility of using vacuum membrane distillation for drying an aqueous suspension of polystyrene microparticles (7 μm; 10 wt%) has been investigated by testing five configurations (VMDr), based on flat and fiber microporous hydrophobic membranes with 0.2 μm pore size [17]. Specifically, tests on three fiber and two flat membrane modules were carried out by both keeping moving the feed (through its recirculation inside the module or the rotation of the module-self) and leaving it in static conditions (in this case the feed was lying on the membrane surface without any movement/agitation). In all proposed designs, one side of the membrane was in contact with the feed to treat, while the other side was kept under vacuum. The membrane was, then, acting as a barrier for both the liquid stream (thanks to its hydrophobicity) and the solid particles contained in the feed (thanks to its pore size that prevented the passage of all material of bigger size). Due to the difference in vapor pressure across the membrane, the liquid water evaporated at the feed-membrane interface, migrated as vapor through the dry membrane micropores and finally condensed outside the module. At the feed side, the vapor pressure is a function of the temperature at the membrane surface (T_m), that is, generally, lower than the bulk value (T_b), due to the temperature polarization phenomenon (heat transfer resistance in the boundary layer). Moreover, during the drying process, a further temperature decay can be offered by the more concentrated feed close to the membrane surface. Fig. 1(a) shows how the dehydration occurs in a VMDr while in Fig. 1(b) a typical temperature profile is reported.

The study carried out led to the identification of the VMDr configuration to be used for efficiently dry the feed. In particular, the flat membrane module working with the feed in static conditions led to a product with a final solid content of 98 ± 0.5 wt%. Moreover, the permeate produced was completely free of solid particles and, then, the collected water was suitable to be reused. Therefore, further tests were carried out on this VMDr configuration by loading a higher amount of feed inside the module, in order to investigate its production efficiency. In this case, it was found that the dehydration efficiency reduced and it was not possible to reach the same dry residue value, even prolonging the duration of the experiments. The result, that affects the system productivity, was attributed to the higher resistance (due to the higher presence of solids) for both water molecules and heat transfer offered by the more concentrated suspension close to the membrane surface, and it was postulated about the possibility of overcoming this issue, by keeping the feed homogeneous during the experiments. However, no further tests were carried out. Based on these results, and having in mind the importance of providing high productivity for practical applications of the developed unit, the present work aimed at further investigating and optimizing the performance of the VMDr flat configuration previously identified, when working with higher feed amounts. Moreover, the effects of the particle size and of the membrane area on the efficiency of the VMDr, were also analyzed. Finally, the effect of the membrane properties on the trans-membrane flux was theoretically calculated.

2. Materials and methods

2.1. Feed properties

Four aqueous suspensions containing 10 wt% of polystyrene microparticles with different nominal size (0.3–0.5–1–7 μm) were purchased from Magsphere Inc. (USA).

2.2. Experimental set-up

The membrane module was made of stainless steel and presented an external jacket. The control of the suspension temperature was made by both sending hot water in the jacket and placing the module inside a thermostated box. The feed temperature was measured by a thermocouple that was 1 mm away from the membrane surface. The module was equipped with adapters, in order to be able to work with different membrane areas. A flat commercial polypropylene membrane (pore size, 0.2 μm; thickness, 91 μm; porosity, 70%) – Membrana (Germany), was placed in the module in such a way that one side was in contact with the feed suspension and the other side was under vacuum. Higher the feed amount inside the module, higher its height on the membrane surface, hereinafter called “suspension head”. The module was operated with the feed under (a) static conditions; (b) mechanical stirring; (c) air bubbling. Fig. 2 shows the set-up and the three different *modus operandi* of the module.

2.3. Experimental procedure

The drying experiments were carried out at mild feed temperature values (30 °C) and applying a vacuum pressure of 4 mbar. The vapor flux was calculated by weighting the condensed distillate recovered in the trap and dividing the weight by the operating time and the membrane area. The solid content of the treated feed and of the produced permeate was determined by means of a moisture analyzer (Ohaus MB-45).

3. Results and discussion

Experiments were carried out at different suspension heads, particle sizes and membrane areas. The reproducibility of results was ensured by repeating each test for at least three times.

3.1. Effect of suspension head

Tests were made on the cell with a membrane area of 7.1 cm^2, by loading on its upper surface different amounts of the 10 wt% aqueous suspension containing 7 μm polystyrene particles.

Fig. 3 shows the dry residue obtained for 3 and 6 mm suspension heads (H), when the feed was kept in static conditions.

It can be noticed that, by doubling the suspension head, the drying efficiency reduced and the dry residue moved from 98 ± 0.5 wt % down to 94 ± 0.5 wt%. No further increments of the dry residue were obtained in this case, even continuing the test for longer time, as was similarly observed in our previous work [17]. Therefore, static conditions do not allow to obtain the same dehydration efficiency when treating higher amount of feed, due the higher resistance offered for both the water molecules and the heat transfer from the suspension bulk toward the membrane surface.

As reported in the Section 1, a possible way to reduce this resistance can be to work with a homogeneous feed and, then, to remove the “barrier to the transport” offered by the more concentrated portion close to the membrane surface. Therefore, next experiments were carried out by mixing the feed by a stirrer. In particular, in order to reduce the mechanical stress on the particles,