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Membrane emulsification for polymeric materials and sorption studies; and its process engineering

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Membrane emulsification, for the production of oil drops in water of a required and controlled diameter between 10 and 1000 micrometres, is becoming increasingly popular. The technique of membrane emulsification originated in the 1990's for the production of drops usually less than 10 micrometres, and was based on work with glass and ceramic microfiltration membranes¹. The formation of drops greater than 10 micrometres was deemed to be difficult, as the conventional microfiltration technique of crossflow filtration was used to provide the shear at the membrane surface to remove the forming oil drops from the membrane. However, crossflow requires the continual recirculation of emulsion through the membrane module and the larger drops, greater than 10 micrometres, often became damaged in the pump and fittings. Later developments were the introduction of a new type of microfiltration membrane², with controlled pore size and spacing between the pores, and new techniques for generating the shear at the membrane surface that enabled larger drops to be produced, and maintained without risk of subsequent drop breakage³. For laboratory studies, a Dispersion Cell was developed; which provides the shear at the membrane surface using a simple paddle stirrer above the membrane disc within a cylindrical vessel, see Fig 1(a).

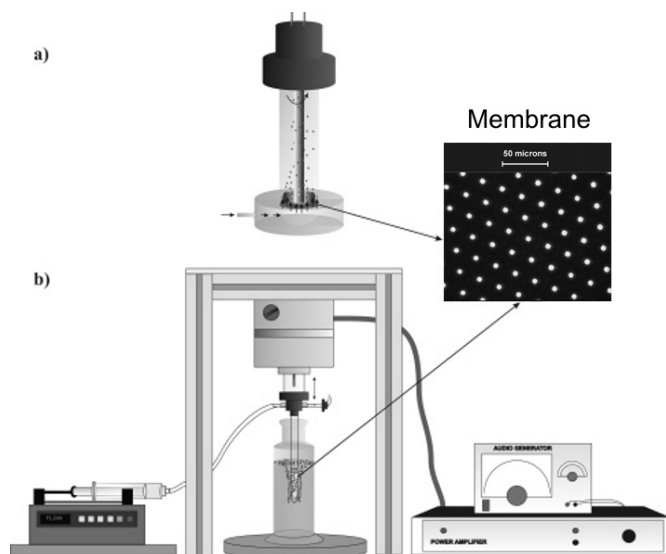


Fig 1 Membrane emulsification equipment

The Dispersion Cell has been used in a number of studies and the drop size, formed by forcing the oil phase through the membrane into an aqueous continuous phase, where the shear from the paddle stirrer breaks the drops off the membrane, has been shown to be reliably predicted from a simple force-capillary balance model. Uniform drops can be produced, with Coefficient of Variations of less than 10%, despite the occurrence of a variable shear field across the membrane surface³. The system has been used to successfully

produce nanoporous polymeric adsorbents, based on a polystyrene matrix, with final bead sizes of between

30 and 40 micrometres, which were designed for the selective removal of middle molecular weight uraemic toxins (size range 05-20 kDa) from blood, whilst minimising the removal of larger blood proteins⁴.

The production of polymeric sorption media using membrane emulsification with the new membrane type that consists of straight-through pores (with no internal structure or tortuous pore channel within the membrane), opens up new possibilities for optimising and designing the resulting polymerised beads. For example, the very low pressure drop required for operation, and the gentle emulsification conditions, means that the production of water-oil-water (w/o/w) emulsions is straightforward. Under normal conditions of emulsification, the generation of the secondary emulsion phase using the primary emulsion (w/o) is problematic

– as the second emulsification stage often leads to the destruction of the primary w/o emulsion. The gentle conditions used by the new membrane type, and the gentle shear conditions in the shear generating systems illustrated in Fig 1, facilitate production of these w/o/w emulsions, that can then be polymerised to produce controlled polymeric release compounds, or other encapsulated products. The example shown in Fig 2 is of both the w/o/w emulsion phase formed and the resulting dry beads under an SEM and is of the biodegradable polymer PLGA⁵, which is approved for use within the human body.

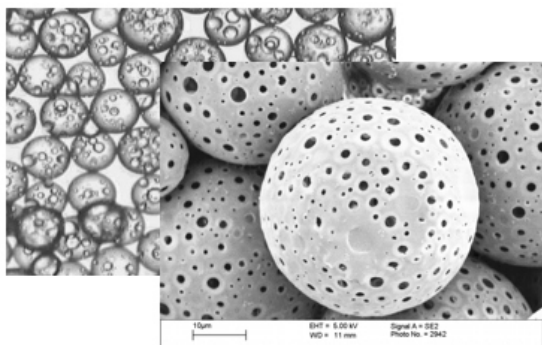


Fig 2 Double emulsion and solidified beads Fig 3 System for 100's kg/h bead production

The simple stirred cell illustrated in Fig 1(a) is clearly not an engineering solution for the generation of larger amounts of beads. For this, the method of generation of shear at the membrane surface is oscillation, as illustrated in Fig 1(b)⁶. This is a scalable method, as the amount of gently oscillating membrane can be easily increased, as illustrated in Fig 3 where a canister membrane is shown, which can be suspended and oscillated from a common membrane hanger for multiple canisters, giving appreciable productivity. Ongoing work includes the production of beads containing oxides (silica, alumina, titania) and other inorganic sorption and catalytic media.

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