**Institution: Loughborough University**

**Unit of Assessment: B12 Aeronautical, Mechanical, Chemical and Manufacturing Engineering**

**Title of case study:**
**The commercial and economic benefits of new technology resulting from Loughborough research into the production of high value particulates and emulsions**

**1. Summary of the impact** (indicative maximum 100 words)

High value particulates and emulsions are now being produced commercially by the process of membrane emulsification using oscillatory technology and uniform pore design membranes developed at Loughborough University (LU). There are only four international companies making commercial quantities of ion exchange particles; two of them use our technology. Smaller chromatography companies have also adopted it, together with other companies making controlled release particles with kg to tonnes per annum throughputs. The technology has been commercialised by Micropore Technologies Ltd, a university spin-out company, and its American subsidiary Micropore Technologies Inc.

**2. Underpinning research** (indicative maximum 500 words)

The research started at Loughborough University in 1994 as an investigation of surface microfilter membranes and the engineering of the processes **[3.1]** and later developed into using the novel membranes and techniques for the process of membrane emulsification **[3.2]**.

The research led to a fundamental revision of microfilter membrane design. New microfilters with a regular array of pores that constitute a 'surface filter' were developed, with diameters of the pores down to 2 µm in width. These membranes do not have tortuous pore channels, or irregular pore openings, and they have very uniform pore size and structure and are sometimes referred to as 'microsieves'. The research also led to the ability to deposit different surface coatings on the membrane to enhance performance and in particular to avoid surfaces that could result in fouling **[3.3, G3.1]**. In addition, surface treatments and operating strategies also provided ways to enhance the membrane operation for both filtration **[3.4]** and membrane emulsification applications **[3.5, G3.2]**.

The application of the membranes to the process of membrane emulsification yielded high throughput production, producing emulsions and particles at a scale in excess of simple laboratory investigation. The process also provided opportunities for 'engineering' the particles in terms of their structure and how the latter could be enhanced for specific applications **[3.6, G3.2]**, e.g. the production of inorganic ion exchange particles for nuclear decommissioning **[G3.3]**. Further research within the Group was directed at investigating novel operating techniques, rather than the conventional method of simple crossflow operation. The new techniques had particular advantages when producing particles with diameters greater than 20 µm, which are required for process-scale ion exchange, chromatography and subcutaneous controlled release depots. These methods have been patented and are now being published **[3.2, 3.6]**. The Group is also active in 'wetting' phenomena research, which is critically important for membrane emulsification **[G3.2]**. Elements of the underlying technology have been published in internationally leading journals **[3.1, 3.2, 3.6]** in the field of Chemical Engineering.

In addition to working with a number of collaborating companies propagating the research results (see s5), in 2003 the Loughborough University spin-out company Micropore Technologies was incorporated to further commercialise the work.

The key researchers who started the work using EPSRC funding **[G3.4]** were Professor Richard Holdich (LU, 1986 to present) and Dr Iain Cumming (LU, 1992 to 2009, when he retired). Additional work on surface modification of the membranes, and novel operating techniques, for generation of the shear at the surface of the membrane was started in 2002 and involved Professor Starov (LU,

1999 to present), whose expertise includes that of surface wetting phenomena. Further studies and developments to the membrane emulsification process took place after Dr Goran Vladisavljevic (LU, 2006 to present) joined the team. Researchers at LU involved with the projects were: Drs Kosvintsev (Research Fellow 2000 - 2006), Zhdanov (Research Associate, 2005 - 2007) and Stillwell (Engineering Doctorate, 2004 - 2009), all of whom obtained initial employment with Micropore Technologies, and Dragosavac (PhD student, 2008 - 2011) who joined the academic staff at LU in 2011.

**3. References to the research** (indicative maximum of six references)

**3.1 Holdich, RG, Kosvintsev, SR, Cumming, IW and Zhdanov, S, "Pore design and engineering for filters and membranes", 2006, Philosophical Transactions of The Royal Society A: Mathematical, Physical and Engineering Sciences, 364(1838), 161-174, ISSN 14712962. DOI: 10.1098/rsta.2005.1690 (Journal Impact Factor 3.11)**
**3.2 Holdich, RG, Dragosavac, MM, Vladisavljevic, G and Kosvintsev, SR, "Membrane Emulsification with Oscillating and Stationary Membranes", 2010, Ind. Eng. Chem. Res, 49 (8), 3810-3817. DOI: 10.1021/ie900531n (Journal Impact Factor 2.21)**
**3.3** Patent: GB2385008(B), Surface Microfilter; Holdich, RG and Cumming, IW; Priority Date 07 Feb 2002
**3.4** Patent: GB2429938(B); Filtering Dispersions; Holdich, RG, Kosvintsev, SR, Cumming, IW; Priority Date 27 July 2005
**3.5** Patent: GB2467925(A); An apparatus and method for assisting vibrating membrane emulsification; Holdich, RG; Priority Date 19 Feb 2009
**3.6 Dragosavac, MM, Vladisavljev, GT, Holdich, RG, and Stillwell, MT, "Production of Porous Silica Microparticles by Membrane Emulsification"; 2011, Langmuir, DOI: 10.1021/la202974b (Journal Impact Factor 4.19)**

The key grants that led to these impacts are:
**G3.1** PI: Holdich, Professor RG, CI: Cumming, Dr IW; Surface microfiltration with slotted media; EPSRC: GR/N05697/01; 01 November 2000 to 31 October 2002; Value £94,597
**G3.2** PI: Starov, Professor VM, CI: Holdich, Professor RG; Superspreading: self-organization at the nano-scale; EPSRC: EP/D077869/1; 20 June 2007 to 19 December 2010; Value £347,839
**G3.3** As a partner in the EPSRC project EP/F055412/1; Title: DIAMOND: Decommissioning, Immobilisation And Management Of Nuclear wastes for Disposal; Principal Investigator: Biggs, Professor SR (Leeds University); 01 July 2008 to 30 June 2012; Value £4,226,934
**G3.4** PI: Holdich, Professor RG, CI: Cumming, Dr IW; Dispersed oil separation from water using surface filters with imposed fluid rotation; EPSRC: GR/K01643/01; 01 October 1994 to 30 September 1996; Value £93,155

**Evidence on the quality of the research**
The underpinning research in s2 was *original* and *rigorous*, as evidenced by the papers **3.1**, **3.2** and **3.6**, all of which are published in high quality journals, and the importance of the research is indicated by the competitive award of funding totalling some £4.8M since 1994. Further evidence is the fact that international groups are now using our techniques and collaborating with our Loughborough Group. Staff from universities in **France** (*Preparation of Liposomes: A Novel Application of Microengineered Membranes – From Laboratory Scale to Large Scale*, A Laouini, C Charcosset, H Fessi all from Université Claude Bernard, Lyon, France, RG. Holdich, GT Vladisavljević both LU; paper accepted for publication in *Colloids and Surfaces B: Biointerfaces*, Journal Impact Factor 3.55), **Italy** (*Microencapsulation of oil droplets using cold water fish gelatine/gum Arabic complex coacervation by membrane emulsification*, 2013, E. Piacentini, L. Giorno both Institute of Membrane Technology, University of Calabria, Italy, M. Dragosavac, G.T. Vladisavljević, RG. Holdich, *Food Research International*, **53**, 365-372, Journal Impact Factor 3.38 and **Turkey** (*Production and evaluation of floating photocatalytic composite particles formed using Pickering emulsions and membrane emulsification*, 2012, RG Holdich, M Lazrigh, G Shama and IY Ipek Ege University, Izmir, Turkey, *Industrial and Engineering Chemistry Research*, **51**, 12509-12516, Journal Impact Factor 2.21) have spent several months at LU investigating novel aspects of the use of the technology for new formulations and applications.

**4. Details of the impact** (indicative maximum 750 words)

There are now a number of commercial products being made employing technology derived from the underpinning research, as cited in s2 and s3. They include ion exchange resin particles, chromatography particles, controlled release pharmaceutical particles and food emulsions (including low fat and low glycaemic index chocolate).

The production of highly uniform particles (with good control of size and structure) for these applications using a single capillary, or microfluidic type of contactor, was demonstrated at the laboratory scale many years ago, but with a very low throughput of less than a few grams per day. Commercially relevant throughput required kg, or even hundreds of kg quantities per day. The techniques developed at Loughborough University (LU) are based on a very uniform membrane medium and oscillatory generation of shear at the membrane-continuous phase boundary *and they enable commercially relevant throughputs to be achieved for these different markets.*

The formation of a university spin-out company, Micropore Technologies Ltd, in 2003 was followed by the formation of Micropore Technologies LLB in the United States of America in 2005 and then Micropore Technologies Inc in 2011 **[5.1,5.2]**. The technology commercialised by Micropore is currently being used in numerous applications within Europe and the USA, for example ion exchange resins by Purolite International and chromatography products by Polymer Laboratories (later Agilent). In addition, controlled release pharmaceutical products based on the LU technology are currently in laboratory trials within the UK and continental Europe, as this is a market with considerable lead time for validation of the products.

During the period from 1st Jan 2008 to May 2013, Micropore has employed at any one time 3 full-time and up to 7 part-time staff; the full-time posts are all PhD level engineers (and all graduated from Loughborough University). The part-time staff have roles ranging from accounting support to manufacturing and engineering design services. The company has been profitably trading (except one year) since its incorporation and is growing organically; the founder shareholders still own well over 50% of the company equity and the University owns the rest. Some personnel have moved on from Micropore to senior positions within client companies involved in membrane emulsification processes, further spreading the impact of the research **[5.3, 5.4]**. During the period covered by the REF, Micropore has had a turnover of over £1M with ~30% growth over the last two years and clients including: Airbus, Arla Foods, Dow Chemical Co, Mars Chocolate and Purolite International. The CEO of Micropore has provided a letter with details of the top ten clients for the company, as well as revenue figures as filed with Companies House **[5.5]**.

In summary, the research that we have cited here has had substantial commercial and economic benefits in the food, analytical, chemical and pharmaceutical industries in Europe and the USA, including employment benefits directly in a UK spin out company. A measure of the impact of our research is that of the only four international companies making commercial quantities of ion exchange particles, two of them use our technology.

**5. Sources to corroborate the impact** (indicative maximum of 10 references)

The following sources can be made available at request:

**5.1** http://www.micropore.co.uk/
**5.2** http://www.microporetech.com/index.htm
**5.3** Statement from the Site Manager of Agilent Technologies at Church Stretton supporting the use of the techniques developed in the research in the production of chromatography particle products.
**5.4** Statement from the Research Director of Purolite International supporting the use of the techniques developed in the research in the production of ion exchange particle products.
**5.5** Statement from the CEO of Micropore Technologies Ltd. supporting the spin out company turnover, growth and top ten clients during the REF period.