EDITORIAL :

Prof. R.J. Neufeld
Queen's University, Kingston Canada

Dear colleagues and friends,

1991-Montréal, followed by Cachan, Brussels, Québec, Potsdam, Barcelona, Easton, Trondheim, Warsaw, Prague, Strasbourg, Vitoria-Gasteiz, Kingston, Lausanne, Vienna, Dublin, Groningen, Porto and now Amboise-2011.

What a roller coaster ride we have had! I for one get dizzy when I think of what has been accomplished by this research association over the past two decades.

Twenty years of International Conferences and add to that COST840 and COST865 action workshops, European/Canadian Space Agency topical team, Industrial Symposia (15 so far, next planned for San Antonio in March 2011), Training Schools.... and countless collaborations, friendships, networks, many glasses of wine and memorable times together, and you have the makings for 20 very successful years of association through what we have called affectionately, the BRG. Worth celebrating!

As I reflect on the past 20 years, a key question that comes to mind is - what has the BRG done for us individually and as a community? The answer for me is clear:

1. BRG through workshops and networking opportunities has built community. This has become my research community of choice as it provides opportunity to develop friendships and collaborations with the top people in our field.
2. My research network through the BRG is world-wide and includes a high level of industrial association.
3. The BRG has defined my field of bioencapsulation, has built credibility and recognition around this discipline, and nurtured development of the technology.
4. BRG connections have enabled valued research collaborations, funding support and venues for research missions at home and abroad.
5. Our association has identified and supported our industrial community through highly successful industrial symposia and through the participation of industrial colleagues in our conferences.
6. Funding opportunities for graduate students and young researchers to attend and participate in our conferences, has provided life-changing experiences for our young colleagues and enriched them, and in turn us, professionally.
7. The BRG has developed dynamic leadership that has been a tremendous and valued support to us all.

Finally, a word needs to be said about our fearless and tireless leader. We owe a debt of gratitude to our founder and President and all round macrospheric nice guy. We have been enriched, encouraged and supported by his dynamic leadership, and by his vision for growing our association.

It will be a terrific year to join us in Amboise, France as we raise our glasses to toast our association. Happy 20th birthday BRG. What will the next 20 years look like?

Best regards,

Ron Neufeld

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14th Industrial Symposium and 5th Trade Fair on Microencapsulation

San Antonio, Tx, USA - March 7-9, 2011

Symposium organization

- Conference Program : 10 lectures of 45 minutes from the leading experts will cover a large scope of the encapsulation field. The selection mixes senior scientists with an understanding of encapsulation processes, with experienced industrials for well established practical applications.

- Exhibition : a broad state-of-the-art showcase presenting R & D services, Equipment & Tools, Material & Chemicals, Established Techniques in the realm of microencapsulation ...

- Technology Trade Fair : based on your own pre-selection among the list of participants, your optimized personal agenda may include up to 20 one-to-one 40 minute appointments. Coffee-breaks, exhibition, and lunch-times give you additional opportunities to establish new contacts.

Conferences

- Encapsulation: Overview on Technologies and Applications - Prof. Denis Poncelet (Oniris and Capsulae, France)
- Precision Particle Fabrication - Prof. Cory Berkland (Kansas Univ. and Orbis Bioscience, USA)
- Applications of Microencapsulation in the Design and Development of Pharmaceutical Products - Prof. James McGinity (University of Texas, USA)
- Regulatory and Safety Considerations for Nanoencapsulated Bioactives - Dr. Bernadene Magnuson (Cantox, USA)
- Improving food using encapsulated ingredients - Dr. Marc Meyers (Meyers Consulting LLC, USA)
- Encapsulated probiotic bacteria for food - Claude Champagne (CRDA, Canada)
- Controlled release in consumer application - Ron Versic (Ronald T. Dodge Company, USA)
- Application of microcapsules in cosmetics - Teresa Virgallito (MicroteK Lab., USA)
- Contribution of microencapsulation to a durable agriculture - Dr. Natarajan Balachander (Landec, USA)
- Novel encapsulation process: perspectives and future - Dr. James Oxley (SwRI, USA)

They are already registered ... join them !

More information at : http://bioencapsulation.net/2011_San_Antonio

Review : Laboratory scale spray drying of inhalble drugs

Dr. Cordin Arpagaus, Dr. Nina Schafroth, Marco Meuri (BÜCHI Labortechnik AG)

INTRODUCTION

The pharmaceutical industry addresses a number of demands on novel respirable particulates, which from a process technology perspective can be broadly categorized into the areas of: performance (e.g. total/local lung deposition, immediate versus controlled release), processing (e.g. achieve flow properties) and stability (e.g. physical/chemical stability and activity).

A new trend in pulmonary drug delivery is to move from liquid or pressurised formulations to dry powder inhalation formulations. This, in part, is due to the advantages of dry powder systems, including breath-actuated inhalation, limited coordination requirements, no propellant requirement and short treatment time [1].

Spray drying is a simple, rapid, reproducible, economic and easy to scale-up production process [2] that has been intensively studied for pharmaceuticals and excipients for pulmonary drug delivery in dry powder inhalation systems [3, 4]. It has the potential to generate highly dispersible powders for inhalation in the range from 1 to 5 µm size with a particle morphology that can more easily be influenced compared to for example jet milling [5].

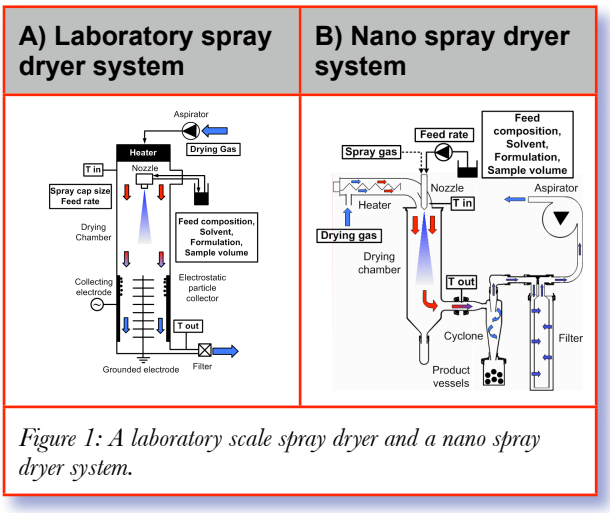

Figure 1: A laboratory scale spray dryer and a nano spray dryer system.

This study reports a review, regarding research work on particles for inhalation that have been published in the RDD proceedings database, using laboratory scale spray dryers (Figure 1).

A laboratory scale spray dryer (Figure 1A) is an instrument to perform spray drying processes up to 1 litre of water or organic solvent per hour. Typically, powders with 2 to 25 µm particle size can be generated by means of a two-fluid nozzle type. To follow the complete drying process from the spray generation down to the powder collection vessel laboratory scale spray dryers are normally made of glassware.

The powder collection is provided by a glass-made cyclone separator, which works by centrifugal forces and virtue of inertia of the solid particles. Adjustable process parameters are the inlet and outlet gas temperature, the drying gas flow rate; the sample feed rate and the spray gas flow enabling scaling-up the process to kg scale.
A nano spray dryer (Figure 1B) is based on a new spray drying concept. The drying gas enters the apparatus from the top where it is heated to the set inlet temperature, flows then slowly through the drying chamber, and exits the spray dryer at the bottom outlet.

The generation of droplets is based on a piezoelectric driven actuator, vibrating a thin, perforated mesh in a small spray cap. The spray mesh features precise micron-sized holes generating a mist of finest aerosol. Millions of fine droplets are ejected with a very narrow distribution. These droplets are dried into solid particles in submicron scale (< 1 µm) and are collected by electrostatic charging and subsequent deflection to the collecting electrode.

LITERATURE REVIEW

A search query in the available RDD (Respiratory Drug Delivery) online proceedings database with the key word “spray drying” revealed 53 hits. Figure 2 visualizes the distribution of these published papers over the last years. It seems that the full potential

of the spray drying process for dry powder aerosols has not been fully exploited yet. Spray drying has become a well established technology in pulmonary drug delivery.

The literature review showed breakthrough R&D innovations in the field of respiratory drug delivery with key information about available spray drying parameters and conditions (summarized in Table 1). Spray drying applications focused especially on anti-asthmatic drugs [2, 5-9], antibiotics [1, 9-12], proteins, such as insulin [13-15], bovine serum albumin [16] or human serum albumin [17], antibodies [18] and tuberculosis vaccine [19].

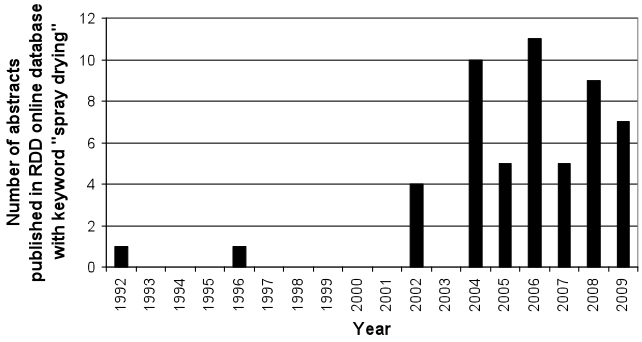

Figure 2: Number of abstracts published in the RDD online database (www.rddonline.com, visited January 8, 2010 with key word "spray drying" (total 53 abstracts)

Various excipients were applied to stabilize drugs during formulation, predominately mannitol [13, 14, 17, 18, 20], poly(lactic-co-glycolic-acid) PLGA [8, 10, 19, 21], lactose [5, 8, 16] and chitosan [7]. SEM photographs of the spray dried powders exhibited mostly spherical shapes with corrugated surfaces, resin-like or even hollow structures, depending on the substance material and drying conditions.
The produced particles were in the respirable size range with roughly 1 - 5 µm aerodynamic diameters. High fine particle fractions were achieved, ranging from 30 - 60% [6-8, 16] to over 85% [13]. Inhaler emitted powder doses of over 90% were reported [2, 7, 8]. Amorphous powders were typically generated due to the short drying time in the laboratory scale spray dryers [3, 22]. Aerosolized powder clouds with maximal volume concentrations of up to 35 ppm particles in air were achieved [20].

High fractions of potentially inhalable aerosol particles of antibiotic cefotaxime sodium were measured for spray dried formulations [23]. Deagglomeration of spray dried protein formulations was possible [17]. High powder dispersibility of spray dried powders was explained by their spherical shape and therefore smaller surface contact area [5].

Particularly, high values of respirable fractions were found for insulin because of the spray dried particle size [13]. The capability for inhalation with relatively high drug loading was shown, for example by incorporation of terbutaline sulphate nanoparticles (an anti-asthmatic drug) into micro particles [2]. Physically and chemically stable non-cohesive spray dried particles, with small aerodynamic diameters were designed to be efficiently delivered as a dry powder aerosol [11]. Spray drying produced powders with satisfactory

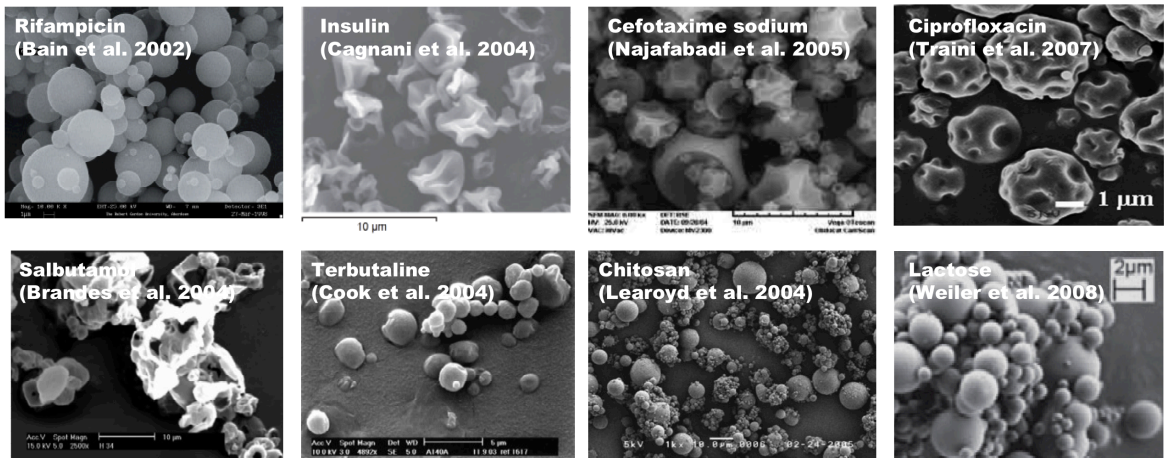

Figure 3: SEM photographs of inhalable spray dried powder from literature.

biochemical stability upon formulation; although with less efficient aerosol properties [24]. Sustained release of highly dispersible amino acid leucine incorporated PLGA powders was exhibited over several days [8].

CONCLUSIONS

Spray drying is a very useful technique to produce inhalable dry powders with predetermined specifications. There is significant research activity in dry powder aerosol formulation to treat several diseases including asthma, tuberculosis, diabetes and bacterial infection in the lung. Spray drying offers great potential to these applications because of the easy achievement of the accepted optimum size range for locally acting inhaled drug particles (about 0.5 - 3.3 µm which represents deposition in the lung alveoli).
The key benefits of this technology are the possibilities to control the size and morphology of the particles under a relatively gentle processing method. Indeed, this method has been proven for the production of heat-sensitive materials such as protein based drugs.

While the traditional bench-top spray dryers have been shown capable tools for the laboratory aim production of respiratory sized particles, the area of process technology is ever-evolving. Nano spray drying (Figure 1) offers new possibilities in the field of laboratory scale spray drying and eliminates some weak points of traditional spray dryers; including increased recovery (up to 90%), small quantity production (100 mg amounts) and highly definable particle size ranges (300 nm - 5 µm) [25, 26, 27, 28].

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Table 1: Literature review of spray dried inhalable products

Drug and application	Particle size, shape, yield, fine particle fraction (FPF) and emitted dose (ED)	Reference and institution
Terbutaline sulphate (asthma drug)	Spherical particles, 3.7 µm size, throat impaction 23.9 %, ED 93%, FPF 46%	Cook et al. 2004 University of London School of Pharmacy, UK & AstraZeneca, UK
Terbutaline sulphate (asthma drug)	1 - 15 µm particle size range, yield 78%, FPF around 40%, ED > 90%	Learoyd et al. 2006a Aston University, Birmingham, UK & Pfizer, UK
Salbutamol sulphate (asthma drug)	Hollow to porous particles, reduced agglomeration tendency compared to jet-milled powders, 40% drug load, FPF 30 - 60 %	Brandes et al. 2004 Christian Albrecht University, Germany
Salbutamol sulphate (asthma drug)	Spherical particles, 0.25 - 3.0 µm, yield 74 %, FPF < 40 %, ED > 90 %	Learoyd et al. 2006b Aston University, Birmingham, UK & Pfizer, UK
Salbutamol sulphate (asthma drug)	Spherical particles, 3.2 µm , FPF around 70 %, dispersion factor around 40 % for 40 L / min	Weiler et al. 2008 Johannes Gutenberg-Univ. Mainz & Boehringer Ingelheim, Germany
Beclomethasone dipropionate (asthma steroid)	Spherical particles, 1 - 5 µm	Cabral Marques and Coimbra 2009 University of Lisbon, Portugal
Insulin (diabetes)	Raisin-like particles, 3.8 µm size, respirable particles FPF > 85 %	Cagnani et al. 2004 University of Parma, Parma, Italy
Insulin (diabetes)	Particle diameter <5.8 µm, sponge-like morphology, suitable for respiratory delivery, FPF 36 - 47%, ED 59 - 81%, dispersability 57-60 %	Najafabadi et al. 2007 University of Medical Sciences, Tehran, Iran & Pasteur Institute of Iran, Tehran, Iran
Insulin (diabetes)	Resin-like morphology, particle size of 4 µm, suitable for inhalation	Maltensen and van de Weert 2008 Univ. of Copenhagen, Denmark
Gentamicin (antibiotic)	Spherical to corrugated shape particles of inhalable size, ED up to 75%, FPF up to 48%	Lechuga-Ballesteros et al. 2004 Nektar Therapeutics, USA
Doxycycline(antibiotic), Ciprofloxacin (antibacterial)	Corrugated particles, 3.7 µm size, FPF < 34 %	Traini et al. 2007 University of Sydney & Monash University, Victoria, Australia

Drug and application	Particle size, shape, yield, fine particle fraction (FPF) and emitted dose (ED)	Reference and institution
Rifampicin (antibiotic)	Spherical particles, 80 % in range 0.3 - 3.0 µm	Bain et al. 2002 Quintiles (UK) Ltd, & Strathclyde Univ., Glasgow & John Moores Univ., Liverpool, UK
Cefotaxime sodium (antibiotic)	Spherical particles, 5.0 µm size, better aerolisation compared to jet milling	Najafabadi et al. 2005 University of Medical Sciences, Tehran, Iran
Tobramycin (antibiotic)	Spherical particles with corrugated surfaces, < 3.0 µm size, yield 60 %, in vitro drug deposition 25 %	Parlati et al. 2008 University of Sydney, Australia
Bovine Serum Albumin (protein)	Corrugated particles, 5.4 / 12.8 µm size, FPF 43.4 %, recovery of drug after inhalation >95 %	Li and Seville 2008 Aston University, Birmingham, UK
Human immunoglobulin (antibody)	No change in secondary proteins structure	Schüle et al. 2004 Univ. of Munich, Boehringer Ingelheim, Germany
Immunoglobulin and Human Serum Albumin	Spherical particles, 2.5 µm (HSA), 4.4 µm (IgG1)	Zimontkowski et al. 2005 University of Bonn & Boehringer Ingelheim,Germany
Proteins secreted by mycobacteria	1.95 µm particle size, yield 53.9 %, activity > 93 %	Garcia-Contreras et al. 2004 University of North Carolina, USA

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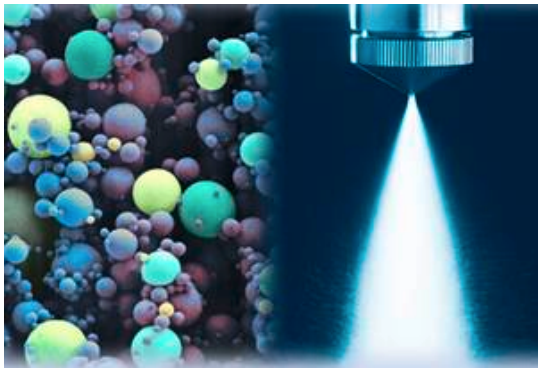

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Conferences

XIX International Conference on Bioencapsulation

Amboise, France - October 5-8, 2011

1991-2011 : twenty years of our association, one generation! We decided to make of our 20th anniversary an exceptional event.

Our annual international conference will take place in one of the most beautiful touristic area of France, the Loire Valley.

Conference and accommodation will be both located in an charming castle-like resort, near to the beautiful town of Amboise. Benefit from those 3 days to exchange on bioencapsulation with other experts coming from all over the world, in a professional but friendly atmosphere. Enjoy the French gastronomy and visit the Leonardo da Vinci last castle-house.

Dear Industrial members,
To insure the success of this conference, allowing students and researchers from all over the world to attend, we do need industrial support. You may help us by simply participating, registering as exhibitor or supporting us through grants for the students. We will be happy to give constructive feedback of your contribution by providing large opportunity of collaborations and contacts with potential customers, advertsing in the newsletter ...
Do not hesitate to contact us *contact@bioencapsulation.net.*

More information at : http://bioencapsulation.net/2011_Amboise

South-America Workshop on Bioencapsulation

Valdivia, Chile - April 20-23, 2011

More information at : http://bioencapsulation.net/2011_Valdivia

Conferences

3rd International Conference on Drug Discovery and Therapy
February 7-10, 2011, Dubai, UAE
http://www.icddt.com/

14th Industrial Symposium & 5th Trade Fair on Encapsulation
March 7-9, 2011 - San Antonio, Tx
http://bioencapsulation.net/2011_San_Antonio/

Fluid bed processing
March 8-10 2011, Binzen, Germany
http://www.ttc-binzen.de/cm/index.php?id=218

Granulation & Tabletting
April 5-7, 2011, Binzen, Germany
http://www.ttc-binzen.de/cm/index.php?id=253

South-America Workshop on Bioencapsulation
Valdivia, Chile, April 2011
http://bioencapsulation.net/2011_Valdivia

2nd Symposium on Biomaterials
May 1-8 , 2011, Heraklion Crete
http://www.bionanotox.org/

8th Scientiific & Technical Forum: Innovative Drug Delivery Systems
May 12-13, 2011, Basel, Switzerland
www.pharmatrans-sanaq.com

5th International Granulation Workshop
June 20-22, 2011, Lausanne, Switzerland
http://www.shef.ac.uk/agglom2011

Continuous particle processing
June 7 - 8 , 2011, Binzen, Germany
http://www.ttc-binzen.de/cm/index.php?id=265

3rd PharmSciFair
June 13-17, 2011, Pragues, Czech Republic
http://www.pharmscifair.org/

Multiparticle Dosage Forms
July 5 - 7, 2011, Binzen, Germany
http://www.ttc-binzen.de/cm/index.php?id=277

Particles 2011: Stimuli Responsive Particles and Particle Assemblies
July 9-12 , 2011, Berlin, Germany
http://nanoparticles.org/Particles2011/

Granulation Course Sheffield on 21-22 July 2011
http://www.shef.ac.uk/agglom2011/course

38th Annual Meeting of the Controlled Release Society
July 30 - August 3, 2011 - National Harbor, Maryland, U.S.A.
http://www.controlledrelease.org/meeting/default.cfm

2nd ISEKI_Food Conference
Aug -31- Sept 2, 2011, Milan, Italy
www.isekiconferences.com/milan2011/

18th International Symposium on Microencapsulation
September 12-14, 2011 - Ankara, Turkey
http://www.microencapsulation2011.org/

Poorly Soluble Drugs Workshop
15 September, 2011, Lille, France
http://www.apgi.org/2011_WS/

XIX International Conference on Bioencapsulation
October 5-8, 2011 - Amboise, France
http://bioencapsulation.net/2011_Amboise

Article : Microencapsulation for chemical applications

Denis Poncelet, Bojana Boh, Gisele Ongmayeb

Microencapsulation consists of embedding an active substance inside a microparticle. The resulting particle may have a different structure such as a solid sphere, a liquid core surrounded by a membrane, a coated solid core or a hydro gel bead. The purposes of encapsulation are diverse. For examples:

- Immobilisation of volatile materials;
- Isolation of reactive ingredients from the matrix;
- Protection of a fragile component during storage or processing;
- Controlled release over time or upon a trigger;
- Material structuration like conversion of liquid in powder;
- Improvement of powder flowing properties.

In fact, involving encapsulation into a process often results from a combination of such reasons. Different types of activesubstances enable multiple applications. As a result it leads that microencapsulation processes deriving from a few initial methods have to be adapted for each specific case.

SHORT HISTORY

The first reference to microencapsulation technologies dates back to the late 1800s (1), consisting of coating by spraying a solution onto particles in a rotating pan reactor. The coating was a first highly concentrated sugar, which was later replaced by a polymer solution. To avoid usage of organic solvents, polymer solutions are often replaced by a latex suspension. Complex formulations were developed combining polymers, surfactants, inert filler materials, and plastifiers to get an adequate release profile. For small particles (less than 1 mm), the pan coating has been generally replaced by a fluid bed to insure better mixing of the particles. Different designs have been proposed such as top spray fluid bed or Wurster process (promoting circulation of particle by inserting a central tube in the reactor). This technology has been especially developed in the pharmaceutical domain (2), but a large scale production of micro-encapsulated chemicals has been allowed by a continuous process.

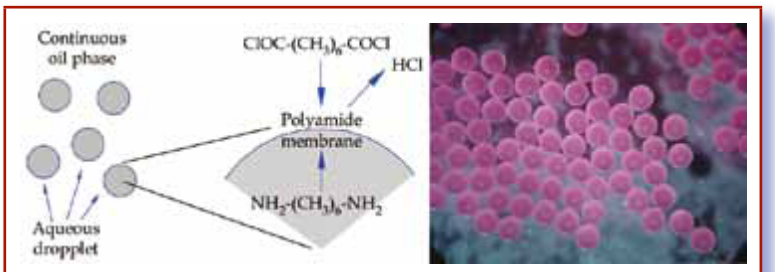


Figure 2. Interfacial coacervation process and resulting capsules © D. Poncelet.

In 1932, spray drying (spraying of a polymer solution in a warm chamber to form small microspheres) has been first sold as a sealed-in volatile flavour in a gum Arabic microsphere (5). When trying to spray dry a fruit juice using isopropanol as a solvent, researchers from Robert and Co detected a strong taste of isopropanol in the resulting powder. They launched further experiments proved that volatile molecules could be entrapped by spray drying. This technology is still largely used in industrial applications, especially for food, but also to

Figure 1. Coating process. A) Pan coater, B) Top spray fluid bed, C) Wurster fluid bed, D) Continuous process (http://www.glatt.de).

produce many other different types of powders. However, many authors consider that the first real industrial application of microencapsulation was initiated in 1954, when the National Cash Register (ncr) introduced the production of microcapsules for the carbonless copy paper. The project was the result of 15 years of research started by Barry Green (3).

Figure 3. Dripping processes: A) vibrated nozzles (http://www.brace.de) B) rotating nozzles (http://www.swri.org).

The first technology for producing liquid core microcapsules was based on coacervation, a phenomenon taking place in colloid systems, where macromolecular colloid rich coacervate droplets surround dispersed microcapsules cores in an emulsion, and form a colloid microcapsule wall, which is then solidified by a cross-linking agent. The main development of ncr carbonless copying paper was based on so-called complex coacervation, combining gelatine and acacia gum colloid solution (4). This process is still largely used today although the use of gelatine is sometimes restricted, and the cross-linking with glutaraldehyde is needed to get strong and hard microcapsules.

Researchers from du Pont have presented in 1959 at the American Chemical Society meeting a series of papers on interfacial polycondensation (6). These papers remain a key reference and lead to an extensive development of microencapsulation by interfacial polycondensation (7). In a process, two or more monomers polymerise around droplets in an emulsion and form a solid polymeric microcapsules wall. One of the monomers is initially dissolved in the aqueous phase, and the other one in a hydrophobic organic solvent, allowing a polymerisation taking place at the droplet interface. The selection of monomers and working conditions
allows to get semi-permeable (also called artificial cells) (8) or impermeable membranes of polyamide, polyester or polyurea. The process was extended and modified to many technological sub-types and chemical variations, such as the in situ polymerisation of formaldehyde with either urea or melamin (9), where both monomers or precondensates are added only to the aqueous phase of the emulsion.

During the 1970's, technologies have been developed based on the solvent removal. After a polymer has been dissolved in a volatile organic solvent, the solution is dispersed into an aqueous phase. The organic solvent is removed by extraction or evaporation at atmospheric or reduced pressure, resulting in the solidification of droplets as polymeric microspheres. The method was developed and widely used in the pharmaceutical field. However, the importance and applicability of microencapsulation of chemicals is reducing due to the cost and the problem of solvent toxicity. More recently, technologies emerged based on dripping either from nozzles (10) or through a spinning device (11). Resulting droplets are solidified by cooling of a melt material, or by gelation of the polymer solution (10). The productivity of such processes has increased over years to allow the production of hundreds to thousands tons per year of capsules with a very narrow size distribution.

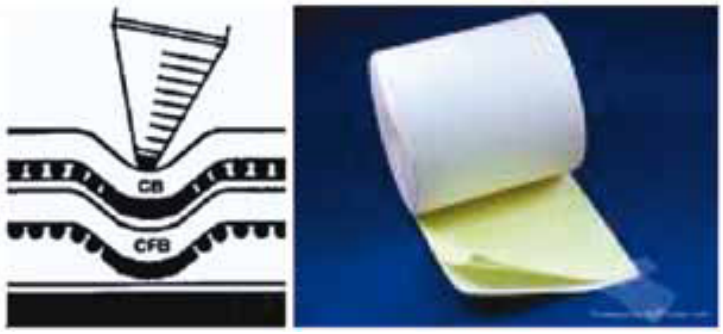


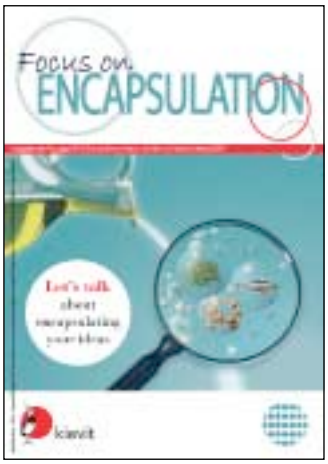
Figure 4. Carbonless copy paper

Several temperature management systems have been developed with microencapsulated phase change materials. For instance, basf has developed a microencapsulated paraffinic phase change material to be incorporated in the building walls within the concrete or plaster matrix structure. When temperature increases, the core material melts, absorbing the energy. When the temperature drops, the material solidifies and gives back the stored energy. The heat capacity of a 15 mm panel with such microcapsules provides buffered temperature behaviour equal to a 7 cm concrete. Phase change material microcapsules may also be used in textiles, e.g. for winter clothes and garments for extreme working conditions. Most actives ingredients included in dishwasher or laundry detergents are included in microcapsules, either for their protection or for a controlled release. This is especially important for the enzymes, where the regulations are very strict, particularly regarding the concentration in the air. Major companies, such as dsm or Genencor, sale encapsulated enzymes in production quantities of thousands tons per year.

Figure 5. Screw coated with encapsulated glue (blue colour).

The structure of the microcapsules became quite complex, with multiple layers containing different active ingredients. Genencor runs the largest plant for microencapsulation consisting of fluid beds with eighty spray nozzles and has developed a processes running for more than 8 hours. Microcapsules are also incorporated in photographic papers, computer screens and other liquid crystal displays, biosensors, crash detector

This article was initially published in



Agro Food Hi-Tech (2009) 20 (2) 6-8
Supplement : Focus on encapsulation

The table of content of this issue was :

- Drusch S., Mannino S. - **Nanostructured encapsulation systems for functional food ingredients**
- Poncelet D., Boh B., Ongmayeb G. - **Encapsulation for chemical applications**
- Fink M., Schleber N., Bollinger H. - Weisbrodt J. et all. Multistage **stabilisation of dried probiotic preparations**
- Meiners J.A., Boquel D., **Applications of multi wall probiotic encapsulation in dairy products**
- Oxley J. - **Analytical technologies applied to micro encapsulation**
- Normand V., Bouquerand P.E. McIver R., Subramanian A. - **How Physical-Chemistry helps in carbohydrate-based encapsulation of Flavours and Fragrances**

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systems, tooth pasts and cosmetics. The number of applications is increasing every day.

CONCLUSIONS

The number of patents on microcapsules has increased quickly during the last three decades. New inventions are often derivates or improvements of previously described techniques. Microencapsulation became a fashion, a hightech field and many products are marketed by claiming the use of microcapsules. Several companies, such as ibm, Kodac, basf, 3M, Rhone-Poulenc, Danisco, dsm, etc. have developed industrial processes and products based on microencapsulation, ranging from a few tons to thousands of tons per year. This contribution could only present an introduction to the large domain of microencapsulation.

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Research Position in France

My university background is the physico-chemistry with a specialization in micro-encapsulation. I have more than 13 years of experience in the formulation of systems of encapsulation, for different customers (Danone, Aventis, Bayer CropScience, Procter&Gamble) in different countries (France, Germany, USA). Projects consisted into the development and optimization of encapsulatin systems like multiple emulsions, nanoparticles and microparticles. Through these different projects, I received awards for Service Merit Award for Customers and award for Spirit of Innovation.

For more information, you can contact me at sgu@new.rr.com

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Article : Creating microcapsules using the TNO encapsulation printer

Kjeld van Bommel

Microencapsulation remains a topic of great interest to the food and pharmaceutical industries as well as many other sectors. Among the various types of encapsulates, core-shell microcapsules are one of the most widely used types. Although the actual application may vary, ranging from taste masking to ingredient protection or controlled release, in the end microcapsules all share the same general characteristics in that they comprise a core consisting of, or containing the active ingredient, and a shell that typically provides some type of barrier function. The fact whether or not a microcapsule performs well generally depends on two main factors: first of all, the nature of the encapsulation material and, secondly, the encapsulation technique or process used to make the microcapsules.

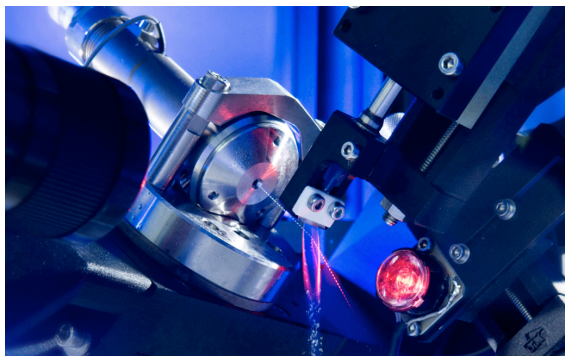

Figure 1: TNO encapsulation printer setup

TNO has developed an innovative new method to make microcapsules by making use of inkjet printing technology. This so-called encapsulation printer (Figure 2) uses a printing head which prints droplets of a liquid through a flowing, liquid screen of an encapsulation material. Upon passing through the liquid film, the original droplet picks up some of the film material and a microcapsule is formed (schematically depicted in Figure 2). It should be noted that the film material not used for encapsulation (i.e. the fraction that flows down after the droplets have passed through the film) can be directly recycled within the setup in order to provide efficient material use.

The printing technology provides excellent process control, resulting in highly monodisperse microcapsules of tunable dimensions with very well-defined shells of tunable thickness. An example of such microcapsules is shown in Figure 3, depicting

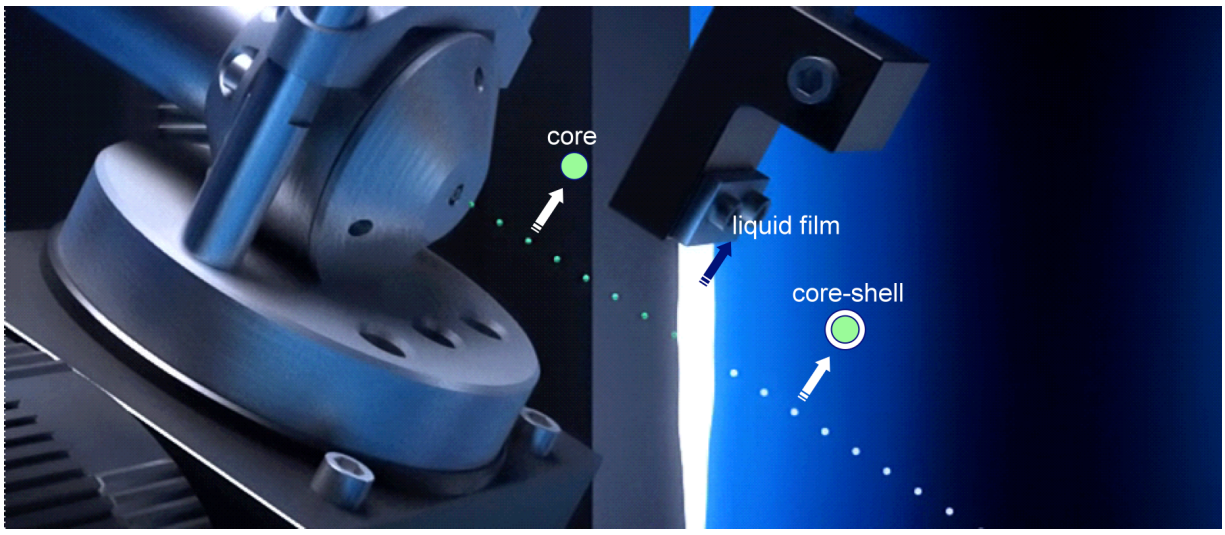

Figure 2: Schematic representation of the microcapsule formation process using the TNO encapsulation printer setup.

monodisperse microcapsules of linseed oil with a carrageenan and gelatin shell. These particular microcapsules have a diameter of 280 µm and a shell thickness of 7 µm (as shown in the right image).

Other microcapsules that have been prepared include flavor burst microcapsules comprising aqueous flavor and colorant solutions encapsulated in a wax; aqueous micronutrient solutions taste masked by encapsulating them in a shell of fat; various oils encapsulated in thin, food grade polymer shells; particle dispersions encapsulated in a food grade polymer shell made via coacervation; and calcium alginate gel particles made by printing alginate droplets through a film of calcium chloride solution. Besides making spherical encapsulates, by adjusting the process parameters, also anisotropic encapsulates can be produced (see Figure 4, right).

With a printing head operating at 20 kHz, the current setup produces 20.000 identical microcapsules per second, corresponding to a production volume of around 200 mL of encapsulates per hour. Both the printing nozzle and film generator can be heated, allowing the use of a wide range of materials, including waxes and fats, polymers, aqueous (or other) solutions, emulsions and dispersions. The separate streams for core and shell material prevent undesired interactions between the two and allow the use of different input temperatures. In the case of other setups this is known to be problematic, as contact between the two materials inside or just after the nozzle will result in undesired, premature heat transfer or reaction, which in turn will either prevent microcapsule formation or may even lead to clogging of the nozzle.

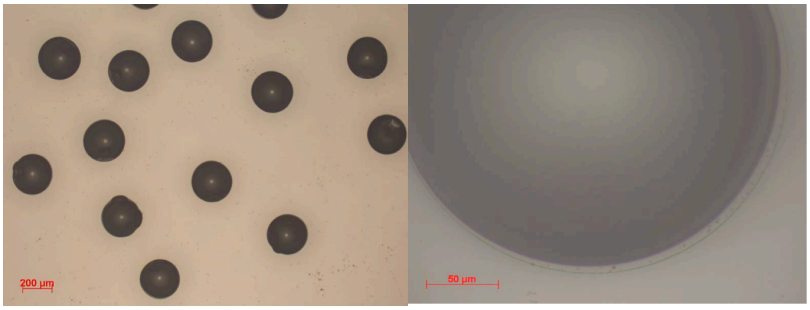

Figure 3: Left: monodisperse microcapsules of linseed oil with a carrageenan and gelatin shell. Right: close up of a microcapsule. Note the thin (7 µm) shell.

In addition, the flight time between the print head and the liquid screen can be used to modify the core material either through solidification (e.g. as a result of cooling), drying (partial or complete), or other methods. Furthermore this technology not only allows the use of liquid cores but may also be applied to solid cores that are shot through the coating screen. Unlike fluidized bed coaters, generally employed for the coating of solids, the TNO technology allows the use of core particles significantly smaller than the approximate 100 µm cutoff used for fluidized bed coating. In addition the applied coatings may be much thinner and, as the encapsulates always remain physically separated during the coating process, they will not aggregate as they do in fluidized bed processes.

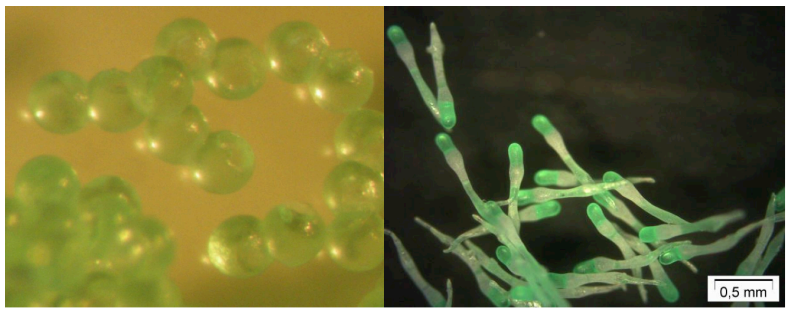

Figure 4: Microcapsules of mint syrup encapsulated by a shell of stearic acid. Left: spherical microcapsules. Right: anisotropic (matchstick-like) microcapsules.

Besides the advantages mentioned above, various other advantages of the encapsulation printer technology exists as can be seen in the inset. Which advantages are most important depends strongly

on the specific microcapsule application. In addition it should be noted that the current encapsulation printer setup is a research setup and is not yet fully developed. Therefore, TNO is currently looking for partners with which to develop this technology further. Such partners include end users from the food, pharmaceutical or other sectors, as well as equipment manufacturers.

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Advantages encapsulation printer

Optimal powders/encapsulates

- very high encapsulation efficiency
- monodisperse particles and shells
- precise dosing of core material (tunable)
- homogeneous product properties (flow, mixing, dissolution)

Material variability

- aqueous, oils/waxes, polymers, solutions, dispersions,
- highly viscous product streams can be used (for core and shell)

Miscellaneous

- converts liquids into powders
- multi-shell microcapsules possible →
- continuous process
- pre- and post-processing possible (drying, gas treatment, …)

Movie : http://www.youtube.com/watch?v=IMlthQXEwag

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