Functionalization of cotton fabrics with natural products

M. Bonet; P. Monllor; E. Bou-Belda; J. Gisbert; P. Díaz; I. Montava
Universitat Politècnica de València, Spain
maboar@txp.upv.es

Abstract

Microcapsules are micrometric spheres, which contain an active ingredient that needs to be protected from some agents. Since Nelson Gordon suggested in the latest 90s that microcapsules could be added to fabrics some research has been focused on this topic, and many active ingredients or shells have been studied.

However, natural products and the development of products and processes environmentally friendly have increased its importance in the last years. Commonly, microcapsules have been based on synthetic polymers for shell and some chemical active ingredients, some natural components have been considered though.

In this work we present the functionalization of cotton fabrics by means of microcapsules from natural ingredients, the active product would be aloe vera for moisturizing skin and the shell would be chitosan, a natural product from the shell of sea food with antimicrobial properties. Depending on the resistance to laundering it is desired, different binders can be used as well. If binder concentration is increased up to 15 g/L microcapsules can remain on fabrics up to 20 washing cycles but touch is altered. If the fabric touch must be unaltered then the resin binder concentration must be 10 g/L and it still will keep hydration behavior on the skin.

We can conclude that conventional garments can be used to treat the skin from any affection if fabrics have been finished properly

Keywords

Microcapsules, cotton, natural products, fibre

Article classification

Research paper

INTRODUCTION

Health care has increased its popularity, as nowadays people are more concerned about beauty. Skin treatment is becoming more and more popular. Hydration is one of the most important factors to keep skin healthy. However, when somebody drinks water it does not go directly to the skin. There are different kinds of skin and its moisture depends on the genes of the person. Furthermore, there are external factors which influence the skin drying it, for example, sun, alcohol, wind, etc.

Skincare products are flourishing day by day and they are offered by a wide range of brands and prices. The active ingredient can be very different even for products in the same brand. Thus, microcapsules can be adhered onto fabrics with the purpose of treating the skin while the cloths are used by the persons.

Microcapsules present an active core, usually liquid, which is protected by means of an external polymer. They are frequently used when the active ingredient needs to be protected from an external agent. For example, an essence is so volatile that when a user wants to breathe its fragrance on a textile it has disappeared. Then if it is encapsulated inside a little capsule, the essence can be released when the user requires it.

The composition of microencapsulated products can be different because they are made of different shell materials and diverse core materials. The core material will define the use, i.e. medicine, food, etc. The cosmetic industry uses fragrances in liquid cores, and although they can be used in aromatherapy, they are not as important as in medicine (Capablanca, 2014).

Microcapsules are used by developed countries into textiles in order to confer added value and allow garments or textiles to be used in non-conventional fields. Microencapsulated products are very common not only in textile but in some fields, such as pharmacy (Szejtli, 2004; Stolnik, 1995; Majeti and Kumar 2002; Orive et al, 2003; Brannon-Peppas, 1999; Kirkpatrick et al., 2002; Magni et al, 2004; Muzzarelli et al., 2004), cosmetics (Vladimir, 2005; Gelabert and Díaz 2001; Eccleston, 1997; Kapuniask and Tomasik 2006; Monllor 2007, Berger et al. 2004.), food (Gouin 2004; Heinzelmann et al 1997; Wegmuller et al 2007; Wibovo et al., 2005; Downham and Collins, 2005; Dewettinck and Huyghebaert, 1999; Wen-taoet al., 2005; De Roos, 2003), insecticides (Boh 2003, Nelson, 2001; Gisbert et al. 2010), adhesives (Giroud, 1995; Aitken et al., 1996), medicine (Muzzarelli et al., 2004, Kapuniask and Tomasik 2006).

Different fields can be combined so that different effects can be added by means of microcapsules. For example the active core can be aloe-vera (hydrating agent and considered as

cosmetic) and the shell can be chitosan. Chitosan (considered as pharmacy or medicine) is a natural product obtained from crustaceous. It is characterised by the antibacterial properties it posses.

Some cutaneous pathologies, such as psoriasis or atopic dermatitis, are characterised by typical effects arising from the mechanical properties of skin (Rodriges et al., 2004). Its treatment can be easy, it can be effective enough if skin is hydrated and kept way from microorganisms.

It is important not only to keep skin soft, but its health is important because it can act as a barrier to prevent some organisms and toxins go trough. The skin hydration level can be determined by the corneometric method, as some studies have demonstrated (Bettinger et al.,.1999.; Pellacani and Seidenari, 2001; Yosipoviitch, and Maayan-Metzger 2000]. The corneometric method is based on the measurement of the capacitance in a dielectric. When changes occur in the hydration of the dermis, the dielectric constant changes and thus causes changes in capacitance.

The aim of this paper is focused on the effectiveness of microcapsules made of natural products. The shell is composed of aloe-vera and the active ingredient (core) is aloe vera. The main objective of these micrpcapsules is to hydrate the skin. It can be useful not only for cosmetic reasons but medical as well.

Chitin is used in wall because it is insoluble, and microcapsules should be broken because of rubbing when fabric is used and then aloe gets in contact with skin and it is hydrated. If aloe is not protected with insoluble agents, it will be lost in for example during washing process, what would reduce their presence and hydration effect.

MATERIALS AND METHODS

2.1.- Materials

Aloe-cored, chitin-walled Color-Center microcapsules were applied to cotton fabric as purchased.

The fabric used was a 100% cotton twill fabric with 210 g/m2, which had been chemically bleached with peroxide in an industrial process.

2.2.- Microcapsules application to fabric.

Commercial microcapsules (30 g/L) were applied onto the surface of the fabric with . In the finishing process a resin was used as a binder with different concentrations: 2,5; 5; 10; and 15 g/L Curing the binder implies a thermal treatment in the form of hot air (120º C).

Application procedure was conducted in a TEPA fulard. The bath treatment for padding comprised of microcapsules with concentration of 30 g/L, and acrylic resin in all baths. was used as a binder with different concentrations: 2,5; 5; 10; and 15 g/L Foulard work was performed at a speed of 2 m/min and cylinder pressure of 1.5 kg/cm in order to obtain a pick-up of around 89–90%. Curing the binder implies a thermal treatment. Samples were thermally fixed in a scale pin stenter at different temperatures for 10 min in WTC BINDER 030.

2.3.- Washing treatment

Washing process was carried out in a Heraus Linitest (Hanau, Germany) for 30 min at 40ºC, in accordance with standard ISO 105 C10. When a cycle was finished, samples were dried on a horizontal surface and the wastewaters from washing cycles were collected to analyse them.

2.4.- *Scanning Electron Microscopy (SEM).*

A JEOL JSM-6300 scanning electron microscope (SEM) was used for surface observation. Textile samples are not conductive so each sample was fixed on a standard sample holder and sputter coated with gold. It was then examined by the SEM with suitable acceleration voltage (10 kV) and magnification.

2.5.- Hydration test.

In order to determine if the hydration result is merely due to the microcapsules presence some placebo tests were conducted. Placebo test was comprised of the cotton fabric without any microcapsule.

Hydration test was carried in two parts for each subject, one for the specific test and the other as a placebo. Some cotton interlock fabric sleeves contained microcapsules and the rest were placebos. Twenty-five subjects suffering from dry skin were selected. They used the sleeves with microcapsules on one side and the others on the other side, each subject didn't know which was each one. Dielectric properties are directly related with skin hydration, and they were determined by capacitance measurements. Hydration was determined by the capacitance test after different periods of time (2, 4 and 6 hours) following a previously published protocol (Marzulli et al., 1976; Berardecasa, 1997; Clarys and Barel, 1997; Clarys et al., 1999;Fluhr et al. 1999). Measurement at 0 hours was established as base line. This test was performed by EVICHispania laboratories.

The results of the corneometric test are expressed arithmetically, based on the average value obtained from all the test subjects, seen in expression 1.

$$\text{Hydration effect (HE)} = T_x / T_0 \quad (1)$$

Where: HE is the hydration effect, and T_0 indicates the corneometric index just before the test. T_x is related to the corneometric value after 2, 4 and 6 hours, as defined in the procedure.

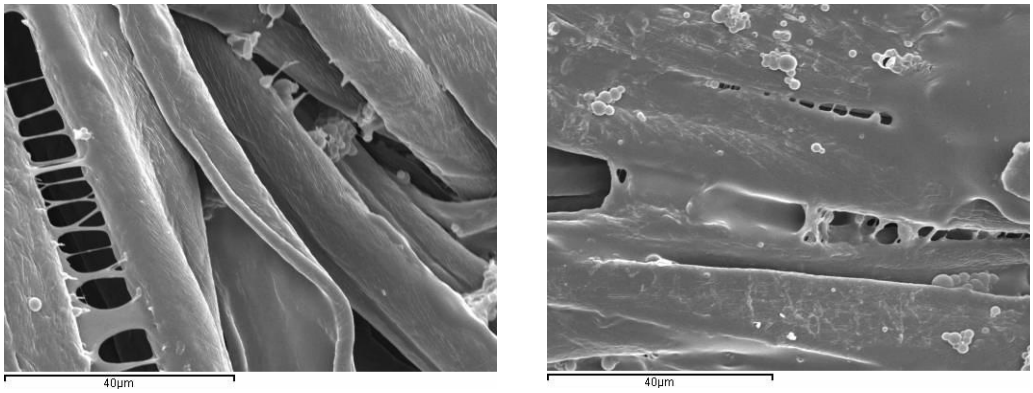
The real hydration effect, or increased hydration effect (IHE) was obtained as the difference between the results from the placebo zone and the treated zone, as seen in expression 2.

$$IHE = (HE)_{\text{microcapsules zone}} - (HE)_{\text{placebo zone}} \quad (2)$$

Where: $(HE)_{\text{microcapsules zone}}$ corresponds to the HE in the zone treated with fabrics not containing microcapsules, and $(HE)_{\text{placebo zone}}$ represents HE in the control zone, in which some fabrics without microcapsule products were tested.

RESULTS

Samples show different concentrations of binder. As it could be expected, at first sight it can be clearly appreciated that when touching the impression on the evaluator is different. Samples with more binder are more rigid than the ones with less acrylic resin. Figure 1 shows the cotton fibres with the higher concentration of binder (15 g/L) and it can be appreciated some thread-like shapes between fibres (Figure 1a) or some thin films (figure 1b).



a) b)

Figure 1.- Cotton samples with 15 g/L of binder.

Higher concentrations of binder reduce the movement between fibres. It is the main cause of the softness reduction when applied on fabrics. When the quantity of resin is reduced up to 2,5 g/L, the presence of resin can be observed (figure 2) but the touch has not been sensitively modified.

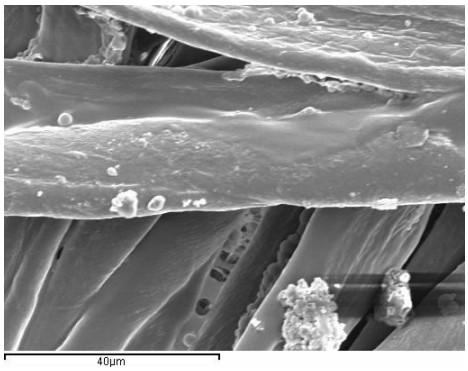


Figure 2.- Cotton samples with 2,5 g/L of binder.

The main objective was to add aloe-vera to the fabric so as to have moisturising effects. In order to determine if it is effective hydration tests were conducted. Fabrics treated with 30 g/L of microcapsules and different resin´s concentration (2,5; 5; 10; 15 g/L). Table 1 shows the results for hydration test after 6 hours of wearing the sleeves.

MICROCAPSULES (g/L)	RESIN (g/L)	IHE (%)
30	2,5	10,9
	5	10,7
	10	10,6
	15	10,6

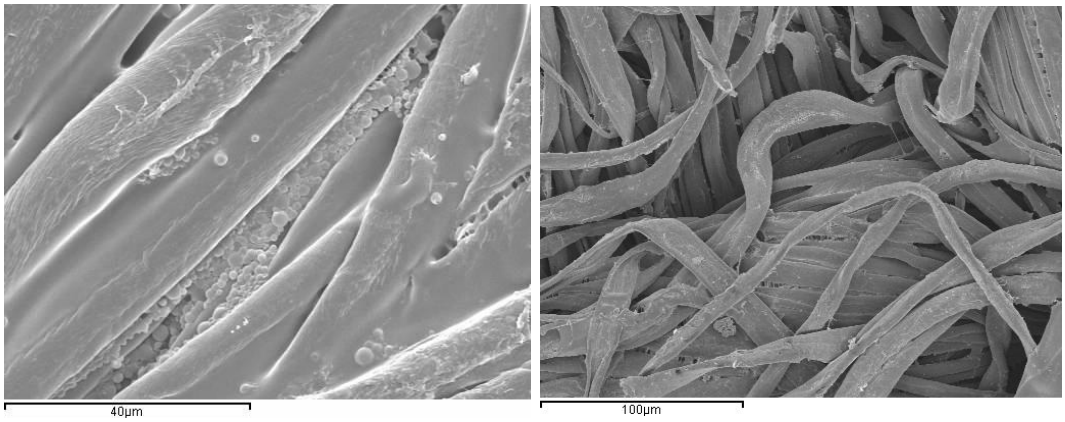
Table 1.- Hydrtation results depending on the resin concentration.

When studying hydration results no significant differences were observed for the hydration. Its value was around 11,7 after 6 hours of exposure to the test as described above.

Resin concentration can be evaluated depending of different parameters, one aspect to consider is the ability of the resin to completely cover the microcapsule. If this happens resin is creating what is called “an iglu effect”, and implies that the resin is protecting so much the microcapsules it is impossible for them to release the active ingredient. It is remarkably the fact that results in table 1 are quite similar if all the samples are compared. As there are no differences, it implies that resin is no covering the microcapsule and prevents it from hydrating the skin. Thus, 15 g/L cannot be considered an excessive concentration for the resin.

However, touch is another parameter to take into consideration. When samples treated with 30 g/L of microcapsules and different quantities of resin, it is noticeable that touch has change if compared with the cotton fabric without any microcapsules. Samples touch is modified slightly form one sample to another, but what has been considered extremely different is the sample which had been treated with 15 g/L. Thus, we consider the concentration of 10 g/L the maximum quantity to apply to a fabric without a high difference.

In order to determine if 2,5 g/L of binder is enough to retain the microcapsules on the fabric surface, some washing treatments were carried out. It was conducted up to 10 laundry cycles. Figure 3 shows the microcapsules after washing.



a) b)

Figure 3.- Fabrics with microcapsules washed 10 cycles. 3a) Fabric padded with 10 g/L. 3b) 2,5 g/L.

It can be clearly seen that it is necessary to increase the quantity of binder as the sample with 2,5 g/L of binder shows few microcapsules. Figure 2b has reduced the enlargement, as it was necessary in order to observe if there were some microcapsules. That means that 2,5 g/L is not enough binder to adhere the microcapsules on the fibre´s surface as the majority of them have been lost. When the test is conducted until 20 cycles, microcapsules on the fabric with 15 g/L still remain on the fabric, the majority of them have been broken as it can be appreciated in figure 4.

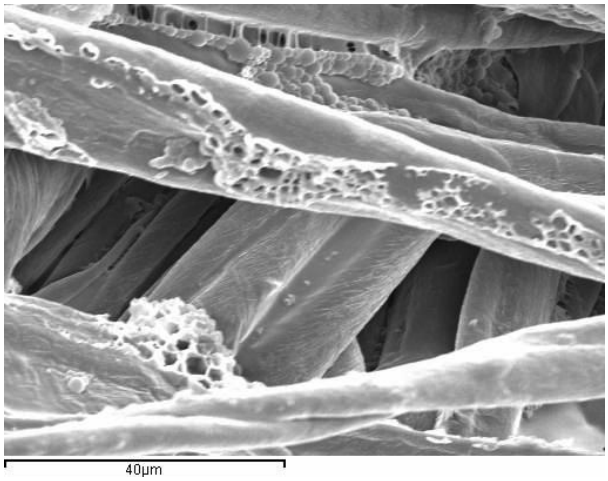


Figure 4.- Fabric with 30 g/L of microcapusles and 15 g/L of binder after 20 washing cycles.

Figure 4 evidences that 15 g/L of resin is enough to keep them on the fibre surface although it cannot be considered useful as the majority of them have not been removed, but they have been broken. Thus, implies there is not hydration effect as the aloe-vera has been spread and gone with washing water. Furthermore, the concentration with higher values showed a difference in touch considerably noticeable. Thus, 15 g/L can be considered as a non-useful concentration for the binder to adhere microcapsules to fibres.

Hydration tests on washed samples were conducted. Table 2 shows the results and as it could be expected they reflect that fabrics which have been washed reduce the hydration of the skin. The reason is that during laundry some microcapsules are unsticked from fibres´ surface. Consequently, when those fabrics were tested on the skin their effect is reduced, as they have not so much microcapsules as the fabrics without any laundry.

MICROCAPSULES (g/L)	BINDER (g/L)	WASHING (cycles)	IHE (%)
30	2,5	10	2,7
30	5	10	6,3
30	10	10	8,7
30	15	10	8,9

Table2. Hydration depending on the binder concentration.

It is clearly observed that when samples have been treated with different concentrations of binder the hydration is modified after laundry. Obviously, the hydration level is lower than the one reached without having washed the fabric (10,7 %). Apparently, there is no significance

between sample treated with 15 g/L and 10 g/L of resin used as a binder. If there is no difference between both concentrations of product, it is useless to apply higher quantities if the result is not considerably improved.

CONCLUSIONS

Microcapsules have been used in textiles for a long time, in this paper cotton fibres have been treated with microcapsules made of natural products. The active ingredient was aloe-vera and the shell was chitosan. At first sight, it has been demonstrated that natural products such aloe-vera, are effective in hydrating the skin despite the fact that it has been encapsulated.

Samples treated with different concentrations of resin show a thread-like shape between fibres. Despite the fact that high concentrations of binder used in this paper (15 g/L) show no "iglu" effect and hydration is the same, touch evidences that there is a big difference and it cannot be accepted if the original touch from cotton needs to be unaltered

Moreover, washing cycles applied to the fabrics reveal that the higher the binder concentration the higher the hydration effect present the fabric as more microcapsules can be observed on the fabric. But when compared with touch it reflects that 10 g/L is the highest concentration for the binder otherwise, touch would be seriously modified.

To conclude, it has been demonstrated that unappreciable quantities of microcapsules on the fabrics can improve the hydration of the skin. This implies that fabrics can be used to apply some compound to the skin. Garments can cover every part of the body and consequently, if they are finished with the proper compounds they can be used to treat any affection on the skin.

REFERENCES.

Aitken D., Burkinshaw S.M., Griffiths J., Towns A.D. Textile applications of thennochromic systems. *Rev. Progr. Color.* 1996, 26 (1), 1-8.

Berardecasa, E., EEMCO guidance for the assessment of stratum corneum hydration: electrical methods. Skin research and technology, 3, pp 126-132. 1997

Berger J., Reist M., Mayer J.M., Felt O., Peppas N.A., Gurny R. Structure interactions in covalently and ionically cross-linked chitosan hydrogels for biomedical applications. *Eur. J. Pharm. Biopharm.* 2004, 57, 19–34.

Bettinger, J.; Gloor, M.; Vollert, A.; Kleesz, P.; Fluhr J.; Gehring, W. Comparison of different non-invasive test methods with respect to the effect of different moisturizers on skin. *Skin Res. Technol.* 5 p. 21-27.1999.

Boh B., Kornhasuer A. Reducing the toxicity of pesticides. *Crit. Rev. Anal. Chem.* 2003, 33 (4), 281-284.

Brannon-Peppas L. Polymers in controlled drug delivery. *Med. Plast. Biomat.*1997, 4, 34-45.

Capablanca, L.; Monllor, P.; Díaz, P.; Bonet, M. Durability of functionalized textiles by microcapsules. MD. Ibrahim H. Mondal (Ed) Chemistry research and applications. Textiles: history, properties and performance and applications. Nova Publisher, New York , 343-354.

Clarys, P., Barel, A.O. In vitro calibration of the capacitance method (Corneomether CM 825 ®) and conductance method (Skicon 200 ®) for the evaluation of the hydration state of the skin. Skin research and technology, 3, pp 107-113. 1997.

Clarys, P., Barel, A.O., Gabard, B. Non invasive electrical measurements for the evaluation of the hydration state of the skin: comparison between three conventional instruments- the Cornemometer ®, the Skicon ® and the Nova DPM ®. Skin research and technology, 5, pp 14-20. 1999.

Dewettinck K., Huyghebaert A. Fluidized bed coating in food technology. *Trend Food Sci. Technol.* 1999, 10 (4-5), 163-168.

De Roos K.B. Effect of the texture and microstructure flavour retention and release. *Int. Dairy J.* 2003, 13 (8), 349-355

Downham A., Collins P. Colouring our foods in the last and next millennium. *Int. J. Food Sci. Technol.* 2000, 35, 5-22.

Eccleston G.M. Function of mixed emulsifiers and emulsifying waxes in dermatological lotions and creams, Colloids and Surfaces. *Physicochem. Eng. Aspects* 1997, 123-124, 169-212.

Fluhr, J. W. and col. Comparative study of five instruments measuring stratum corneum hydration (Cornemometer Cm 820 and CM 825 ®, the Skicon ®, the Nova DPM 9003 ®, Dermalab ®) Part II In vivo. Skin research and technology, 5, pp 171-178. 1999

Gelabert Y.E., Díaz R. Nuevas alternativas de inmovilización de activos antioxidantes. *Revista cubana de investigaciones biomédicas. Ed. Ciencias Médicas: Ciudad de la Habana (Cuba)*, 2001; Vol. 20(1), pp 70-73.

Giroud F., Pernot J.M., Brun H., & Pouyet B. Optimization of microencapsulation of acrylic adhesives. *J. Microencapsulation* 1995, 12, 389-400.

Gisbert J., Bonet M., Riobo P.M., Monllor P. Insect Repellent Textile. *US Patent* 2010/0183690 A1, 2010.

Gouin S. Microencapsulation: industrial appraisal of existing technologies and trends. *Trend Food Sci. Technol.* 2004, 15 (7-8), 330-347.

Heinzelmann K., Franke K. Using freezing and drying techniques of emulsions for the microencapsulation of fish oil to improve oxidation stability. *Colloids Surf., B: Biointerfaces* 1999, 12 (3), 223-229.

Kapuniask J., & Tomasik P. Lipid microencapsulation in starch. *J. Microencapsulation* 2006, 23, 341-348.

Kirkpatrick C.J., Krump-Konvalinkova V. Unger R.E., Bittinger F., Otto M., Peters K. Tissue response and biomaterial integration: the efficacy of in vitro methods. *Biomol. Eng.* 2002, 19, 211-217.

Magnin D., Lefebvre J., Chornet E., Dimitriu S. Physicochemical and structural characterization of a polyionic matrix of interest in biotechnology, in the pharmaceutical and biomedical fields. *Carbohydr. Polym.* 2004, 55, 437-453.

Majeti N., Ravi Kumar V. Nano and microspheres as controlled drug delivery devices. *J. Pharma. Sci.* 2002, 3 (2), 234-58

Marzulli, F.N., Maibach, H.I., Contact allergy: predictive testing in man. Contact Dermatitis, 2, pp 1-17. 1976.

Monllor P. Caracterización de microencapsulados aplicados sobre materiales textiles. Ph.D. Thesis, Universitat Politècnica de València, Valencia, 2007.

Muzzarelli C., Stanic V., Gobbi L., Tosi G., Muzzarelli R.A.A. Spray-drying of solutions containing chitosan together with polyuroans and characterisation of the microspheres. *Carbohydr. Polym.* 2004,57, 73-82.

Nelson G., Microencapsulates in textile finishing. *Rev. Progr. Color.* 2001, 31, 57-64.

Orive G., Hernandez R.M., Gascón A.R. Cell Encapsulation: Promise and Progress. *Nat. Med.* 2003, 9 (1), 104-107.

Pellacani, G., Seidenari, S."Water sorption- Desorption Test and moisture Accumulation Test for Functional Assesment of Atopic Skin in Children". Acta Demato-Venereologica. Vol 81.n2,. pp 100-103. 2001

Rodrigues, L.M.; Pinto, P.C. Analysis of the influence of hidration of the epidermis on the biomechanical behaviour of in vivo skin. Ars. Pharmaceutica, 45:1; 59-71, 2004

Stolnik S. Long circulating microparticulate drug carriers. *Adv. Drug Del. Rev.* 1995, 65, 45-49.

Szejtli J. Past, present, and future of cyclodextrin research. *Pure Appl. Chem.* 2004, 76 (10), 1825–1845.

Vladimir P. T. Recent advances with liposomes as pharmaceutical carriers. *Nat. Rev. Drug Discov.* 2005, 4, 145-160.

Wegmuller R., Zimmermann M.B., Buhr V.G., Windhab E.J., & Hurrell R.E. Development, stability and sensory testing of microcapsules containing iron, iodine, and vitamin A for use in food fortification. *J. Food Sci.* 2006, 71, 181-187.

Wen-tao Q., Wei-ting Y., Yu-bing X., Xiaojun M. Optimization of Saccharomyces cerevisiae culture in alginate-chitosan-alginate microcapsules. *Biochem. Eng. J.* 2005, 25 (2), 151-157.

Wibovo S., Velazquez G., Savant V., Torres J.A. Surimi was water treatment for protein recovery: effect of chitosan-alginate complex concentration and treatment time on protein adsorption. *Bioresour. Technol.* 2005, 96 (6), 665-671.

Yosipoviitch, G., Maayan-Metzger, A., Merlob, P., Sirota, L.. "Skin Barrier properties in different body areas in Neonates". Pediatrics. Vol. 106. n1. pp 105-108. 2000.

ISBN: 978-989-20-5337-0