

# Effect of Ultrasound Emulsification on Cheese Aroma Encapsulation by Carbohydrates

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The encapsulation of liquid cheese aroma (20%) in different carbohydrate matrices by spray-drying was investigated. Carbohydrate stabilized emulsions have been prepared by two emulsifying methods, ultrasonic or Ultra-Turrax treatments, and have been compared in terms of emulsion stability and encapsulation efficiency. The use of ultrasound is particularly effective to obtain a stable emulsion with maltodextrin as support, which is known for poor emulsification properties. In the same way, the spray-dried maltodextrin microcapsules were more effective for retaining cheese aroma when ultrasound (12.7 g/100 g of dry powder) was used for the emulsification step rather than Ultra-Turrax (10.7 g/100 g of dry powder). In terms of encapsulation efficiency, the best system of cheese aroma encapsulation is obtained using ultrasound for the emulsification step and modified starch as support (94.3%). With this support, the positive effect of ultrasound resulted in a lower microcapsule size and in a higher aroma retention than when Ultra-Turrax was used (83.3%). Studies on the aroma profiles showed changes after encapsulation that depend not only on the nature of the support and the emulsification method but also on the interactions between the aroma compound and the matrix. In terms of flavor quality, the best system of cheese aroma encapsulation is obtained using ultrasound and maltodextrin as support.

**Keywords:** Encapsulation; carbohydrates; ultrasound; emulsification; cheese aroma; retention

## INTRODUCTION

Microencapsulation is defined as a technique for packaging sensitive ingredients in a sealed capsule and is applied for many reasons: to protect the core material (or internal phase) and ensure against nutritional loss, to control the release of the core material, to separate reactive or incompatible components of a formulation, to transform liquids into easily handled solid ingredients, and to mask or preserve flavor (Balassa and Fangler, 1971; Dziezak, 1988). Indeed, flavors are inevitably composed of aroma compounds that are volatile and sensitive and which in many cases are oxidized or hydrolyzed; the use of encapsulated aroma compounds solves many formulation related problems and has stimulated the development of new food products (Bandhari et al., 1992; Reineccius, 1995; Goubet et al., 1998).

Microencapsulation of aroma compounds is realized by processes such as spray-drying, spray chilling, spray cooling, air suspension coating, extrusion, coacervation, inclusion complexing, and freeze-drying (King, 1995). Spray-drying is the choice of dry flavor manufacturers because of its ease and low cost (Reineccius, 1981; REMI, 1998). When spray-drying is used, the retention of aroma compounds is highly affected by the solid content of the dryer and the processing temperature and also by the nature and the performance of the encapsulating support, that is, the emulsion stabilizing capabilities,

film forming ability, and low viscosity at high concentration (Reineccius, 1988; Rosenberg et al., 1990; Goubet et al., 1998). Carbohydrates such as maltodextrin, modified starch, and gum arabic are used extensively in spray-dried encapsulations of food ingredients as the encapsulating support, that is, wall material or carrier (Reineccius, 1991; Kenyon, 1995; McNamee et al., 1998). The emulsifying quality of modified starch or gum arabic allows an important oil retention, whereas maltodextrin has a much lower retention ability but lower viscosity relative to modified starch and so may be used at higher dryer feed solid.

The importance of preparing a fine emulsion prior to microencapsulation of volatiles has been well documented (Risch and Reineccius, 1988; Trubiano et al., 1987; Sheu and Rosenberg, 1995). It has been suggested that retention of volatiles during microencapsulation by spray-drying could be enhanced by reducing the mean particle size of the dispersed core material during emulsification. Emulsifying equipment such as a mixer, Ultra-Turrax, or pressure homogenizer is typically used. Emulsions can be prepared by an ultrasonic apparatus too, which was one of the earliest uses of ultrasound in the textile, cosmetic, pharmaceutical, and food industries (Mason et al., 1996). The emulsions obtained by means of mechanical oscillations at ultrasonic frequencies are known to possess a number of qualitative advantages, which have been described in the literature for a number of years (Neduzhii, 1962, 1965; Li and Fogler, 1978a,b; Reddy and Fogler, 1980; Mason et al., 1996): stable emulsions even without the addition of surfactant are generated with ultrasound. The mean droplet size of the suspensions is smaller within a narrow size distribution compared to that obtained with

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other methods. Because of these two characteristics, emulsions generated by ultrasound may find potential applications in areas where uniform and small particle size is essential, such as in encapsulation.

The aim of this study is to compare in terms of emulsion stability and flavor retention spray-dried aroma cheese powders obtained after emulsification by ultrasound or by a conventional mechanical dispersion using an Ultra-Turrax homogenizer. Using an ultrasound device, the most optimum conditions to obtain stable emulsions with different supports will be determined. Studies on the changes in the aroma profile will be undertaken to understand the effect of support and emulsification method on encapsulated aroma quality.

## MATERIALS AND METHODS

**Wall and Core Materials.** Maltodextrin with dextrose equivalent DE-12 and octenyl succinate starch were purchased from Roquette France (Lestrem). Liquid cheese aroma from Griffith Laboratory (Neuilly, France) was used as core material. The aroma solution was essentially constituted with fatty acids (97%).

2-Heptanone used as model aroma compound to emulsion assays was purchased from Fluka Co.

**Emulsion Preparation.** Wall solutions containing 40–55% (w/w) solids were made in 50 °C distilled water. To get all of the hydrocolloid fully rehydrated, octenyl succinate starch solution was prepared and allowed to stand overnight at room temperature before use, whereas the solution of maltodextrin was prepared 2 h prior to use. Twenty grams of aroma products per 100 g of support (modified starch or maltodextrin) was added to the wall solutions before homogenization. Twenty grams of cheese aroma was equivalent to 17.77 g expressed in butyric acid equivalents. Emulsion assays were realized with samples of 50 mL. Emulsifications were carried out by using an Ultra-Turrax T-25 homogenizer (IKA Janke and Kunke model Bioblock Scientific, Illirch, France) operated at 25000 rpm for 10 min or by using an ultrasonic instrument (Bioblock Scientific model).

The ultrasonic apparatus was a sonifier consisting of a power supply, a sonic converter, and a step-horn transducer. It operated at a frequency of 20 kHz with a maximal power output of 600 W. The power supply had an output control with a dial marked in arbitrary units from 1 to 10. The sound probe was immersed at a constant depth and placed centrally in the emulsion. Emulsions were prepared at sound intensities recorded as power inputs of 120–200 W (levels 4–10). The applied time of the ultrasonic treatment varied to 5 to 20 min.

The Ultra-Turrax and ultrasonic treatments were realized at a temperature of 50 °C in a cell where it was possible to minimize temperature fluctuation by circulation of thermostated water in a double jacket.

**Spray-Drying.** For each experiment, 1.5 L of the emulsions was spray-dried using an APV Anhydro Laboratory spray-dryer (Lab 3, Baker, Evreux, France). The dryer had a maximum evaporation rate of 8 kg of water/h, and it was equipped with a rotary atomizer nozzle. Inlet air at 175 °C and outlet air at 90 °C were used in all cases. The dried microencapsulated products were stored at 4 °C until they were analyzed.

**Analytical Methods. Emulsion.** The size of the volatile droplets dispersed in the emulsion was measured under a light microscope after coloration with eosin (0.5% v/v aqueous solution, Fisher Scientific, Elancourt, France). The microscope (Leica DMLB, Fisher Scientific, Elancourt, France) was equipped with a camera and an image print system (Sony, Fisher Scientific). The particle size was determined on the photomicrographs with a micrometer.

Viscosity measurements were made using a Brookfield viscometer (RVT, Beckman, Gagny, France) equipped with a No.1 spindle (cylinder, 56 mm diameter). The apparent viscosity was measured at a rate of 10 rpm at 50 °C and was taken

as an average value of duplicate readings. The physical stability of the emulsion, that is, creaming and flocculation, was determined by storing samples for 5 days at ambient temperature and recording the moment at which the cream layer was formed.

**Powders. (a) Total Fatty Acids Retention.** As cheese aroma was essentially constituted with fatty acids (97%), the total aroma content of the powders was determined in triplicate by fatty acids titration. Fatty acids were extracted according to the method of Wolff (1993): 2 or 3 g of the powder was dispersed at 60 °C with a magnetic stirrer during 15 min in 10 mL of water. After cooling, 25 mL of a hexane/2-propanol mixture (3: 2 v/v) was added to the suspension, which was stirred for 15 min. Fatty acids amounts were estimated in duplicate by titration with a KOH/ethanol solution (0.5 N) in the presence of phenolphthalein. Results were expressed in grams of butyric acid per 100 g of dry powder; indeed, butyric acid was the major fatty acid (78%) of cheese aroma.

**(b) Moisture.** The moisture contents of the spray-dried microencapsulated products were determined in duplicate via a toluene distillation method (AFNOR, 1985). A 20 g sample of powder was refluxed for 5 h with 100 mL of toluene in a 500 mL boiling flask filled with a Bidwell sterling trap and a water-cooled condenser.

**(c) Aroma Components Profile.** The profiles of aroma components in the powder were verified by means of a solvent extraction method. Powder samples (2–3 g) were dissolved in water (10 mL), hexane (15 mL) and an internal standard (octane) were added, and the mixture was stirred for an optimized time of 30 min in a closed flask to avoid solvent evaporation. Traces of water were removed using anhydrous sodium sulfate before analysis by gas chromatography. The percentage of each fatty acid was calculated on the basis of total fatty acids peak area.

A gas chromatograph (Hewlett-Packard 5890 series II, Bron, France) equipped with an FID and with a software system was used. A polar (BP 10, SGE, Villeneuve St. George, France) silica capillary column, 50 m × 0.32 mm i.d., film thickness of 0.25 μm, was operated with the following temperature program: 40 °C for 10 min, raised at 10 °C/min to 115 °C, held for 10 min, raised at 4 °C/min to 200 °C and then at 10 °C/min to 240 °C. Helium was used as the carrier gas at 1 mL/min, and injections were in split mode (1:10).

**(d) Surface Aroma Determination and Encapsulation Efficiency.** The amount of extractable surface aroma of the dried powder was determined in duplicate by Soxhlet extraction (Risch and Reineccius, 1988). Ten grams of powder was put in an extraction thimble and covered with glass wool. The powder was extracted with 60 mL of pentane for 5 h. An internal standard (octane) was added to the extract prior to evaporation, which was carried out under nitrogen until a final volume of 1 mL. The amount of oil in the samples was determined by gas chromatography with the method previously described. The encapsulation efficiency has been calculated on the basis of a theoretical cheese aroma content of 20% of the solids expressed in grams of butyric acid and by taking into account total fatty acids retention and the surface aroma.

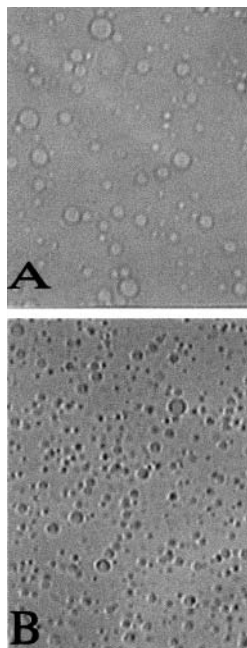
$$\text{encapsulation efficiency} = \left[ \frac{\text{total fatty acids} - \text{surface aroma}}{\text{theoretical cheese aroma}} \right] \times 100$$

**(e) Scanning Electron Microscopy (SEM).** A Philips XL 30 (Philips Electronic Instruments, France) scanning electron microscope was used to investigate characteristics of particle outer structures.

The powder was mounted on stubs using double-stick carbon tape, and the stubs were coated with gold to make the sample conductive. All samples were examined in the backscattered electron imaging mode at an acceleration voltage of 20 kV. Photographs were taken at magnification from 500×.

## RESULTS

**Emulsion Characteristic.** 2-Heptanone was used as model aroma compound to preliminary emulsion



**Figure 1.** Effect of power on the droplet particle size of a modified starch (40%) stabilized emulsion obtained by ultrasound treatment for the same sonication time of 10 min: (A) power level 8, 160–170 W; (B) power level 6, 140–150 W. The figure is reproduced here at 75% of its original size ( $\times 40$ ).

assays. Emulsions, made up of 20% of 2-heptanone per weight and different carriers, have been prepared using an ultrasonic apparatus or a conventional laboratory Ultra-Turrax.

Preliminary studies were undertaken to determine the optimum power and sonication time necessary to obtain a stable emulsion with ultrasonic treatments. Indeed, the emulsion quality depends on the nature of the initial components, on their concentrations and viscosity, and also on the acoustical parameters of the field such as the frequency, the power, and the application time (Neduzhii, 1962; Higgins and Skauen, 1972; Li and Fogler, 1978a).

By changing the sonication time at a constant power level of 6 (140–150 W), the particles size varied. With maltodextrin and modified starch as carriers, the optimum time was 10 min. Indeed, a sonication time of  $< 10$  min produced large particles with a wide distribution, whereas a sonication time  $> 10$  min produced coalescence. In the same way, as the power level was increased to 8 (160–170 W), the droplet size increased and droplet coalescence appeared for the different support (Figure 1). Using modified starch as support, there was very little change in emulsion quality at the two power levels of 4 (120 W) and 6 (140–150 W), whereas with maltodextrin, at a power level of 4, the energy was inadequate to obtain a stable and fine emulsion. Therefore, a power level of 6 and a sonication time of 10 min were applied to the different carriers, and the emulsions were compared to the emulsions obtained by Ultra-Turrax treatment (Table 1).

Using maltodextrin as carrier, the emulsion quality is improved by the use of ultrasound. The size of the majority droplets was 2 times smaller than those obtained using the Ultra-Turrax treatment, but a large size distribution is observed (Figure 2). The emulsion revealed a higher stability against creaming (which appeared only after 12 h) and coalescence than the emulsion produced by the Ultra-Turrax method. With

**Table 1. Characteristics of Emulsions Obtained by Ultrasound or Ultra-Turrax Method**

nature of support	% of support	emulsification method	mean particle size ( $\mu\text{m}$ )
octenyl succinate starch	40	Ultra-Turrax	2.1
		ultrasound	1.8
maltodextrin	50	Ultra-Turrax	8.9
		ultrasound	3.9
maltodextrin/octenyl succinate starch	55	Ultra-Turrax	2.2
		ultrasound	2.3

the two emulsification methods, the modified starch gives in the average oil a similar droplet size of  $\leq 2 \mu\text{m}$ ; there were no perceptible changes in the droplet size, but a narrow size distribution seemed to be obtained; the droplet concentration was stronger and the stability against creaming was increased (appeared after 24 h). With a maltodextrin/modified starch ratio of 5:3 and a solid level of 55%, the mean droplet size ranged from 2.2 to 2.3  $\mu\text{m}$  and was similar to the size obtained with modified starch. Observation with optical microscopy for sizing the particles is subjective. Using eosin as colorant of the hydrophilic phase permitted us to obtain a good characterization of the dispersion state of the particles (the inner portion of the droplet of cheese aroma appears clear), but an electronic counting will permit us to obtain most information.

Similar results were obtained using cheese aroma as core material. In this case, the measurement of the emulsion viscosity has shown that the use of ultrasound resulted in only minor changes in the viscosity of emulsions except with the mix for which the viscosity was highly increased (Table 2). The emulsions stabilized by modified starch exhibited the highest viscosity value of 96 cP, followed by the maltodextrin-modified starch emulsion.

#### Fatty Acids Retention in Spray-Dried Powders.

**Total Fatty Acids Recovery.** The amounts of fatty acids entrapped in the various carriers systems after emulsification by ultrasonic or Ultra-Turrax and microencapsulation by spray-drying were compared in grams of butyric acid for 100 g of dry powder (Table 2). Irrespective of the emulsifying methods, the highest total aroma levels are exhibited by the support systems constituted by in the modified starch, whereas the lowest levels are obtained with the maltodextrin support. The fatty acids of the cheese aroma were highly retained (60–95%) in contrast with previous results, which showed that the retention rates of butyric acid and caproic acid were  $< 7\%$  on freeze-dried carbohydrates (Voilley, 1995). The blending of modified starch with maltodextrin resulted in the increase of aroma retention (from 10 to 18%) due to the increase of the soluble solids at a low viscosity.

For the same support, the use of ultrasound for the emulsification step increased the cheese aroma retention. The increase rose until 10% with the maltodextrin and the modified starch as carrier, whereas no changes were obtained using the maltodextrin-modified starch support.

**Moisture Content.** The moisture content of the different powders ranged from 3.14 to 4.36% and seemed to be independent of emulsification steps but directly correlated with the nature of the carriers.

**Changes in Aroma Profile.** The results of the aroma profile study (Table 3) are expressed as the percentage of each volatile fixed on the carriers. This study showed

**Table 2. Composition of Powders Obtained by Spray-Drying after Emulsification by Ultra-Turrax or Ultrasonic Treatment**

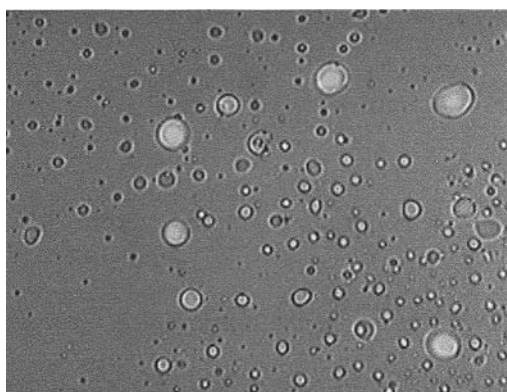
support	emulsification method	viscosity at 50 °C (cP)	% of support	total fatty acids <sup>d</sup> [g (100 g of dry powder) <sup>-1</sup> ]	moisture content (%)
octenyl succinate starch	ultrasound <sup>b</sup>	96	40	16.9 ± 0.50	4.36
	Ultra-Turrax <sup>c</sup>	93	40	15.0 ± 0.81	4.02
maltodextrin	ultrasound <sup>b</sup>	28	50	12.7 ± 0.16	3.90
	Ultra-Turrax	26	50	10.7 ± 0.14	3.88
maltodextrin/octenyl succinate starch (5/3) <sup>a</sup>	ultrasound <sup>b</sup>	74	55	14.6 ± 0.34	3.6
	Ultra-Turrax <sup>c</sup>	55	55	14.2 ± 0.45	3.14

<sup>a</sup> Proportion of each support. <sup>b</sup> Power = 140–150 W. <sup>c</sup> 20500 t/min. <sup>d</sup> Mean ± SD, *n* = 3.

**Table 3. Percentage of Each Fatty Acid (Expressed as a Function of Total Peak Fatty Acid Area) in Liquid Cheese Aroma and in the Different Powders**

fatty acid	liquid cheese aroma <sup>a</sup> (%)	octenyl succinate starch		maltodextrin		octenyl succinate starch /maltodextrin	
		UT <sup>b</sup>	US <sup>c</sup>	UT <sup>b</sup>	US <sup>c</sup>	UT <sup>b</sup>	US <sup>c</sup>
butyric acid	78.8 ± 3.0	50.1 ± 1.62	94.7 ± 0.42	51.6 ± 2.51	77.5 ± 2.81	88.2 ± 1.34	94.0 ± 2.47
hexanoic acid	0.97 ± 0.21	0.74 ± 0.97	<0.01	0.47 ± 0.48	<0.01	0.81 ± 1.15	0.98 ± 0.01
octanoic acid	1.88 ± 0.31	2.45 ± 0.61	0.37 ± 0.52	3.32 ± 1.50	<0.01	1.22 ± 0.72	0.96 ± 0.10
decanoic acid	3.5 ± 0.42	6.9 ± 0.84	1.47 ± 0.46	9.07 ± 3.84	3.9 ± 0.69	3.37 ± 1.08	1.3 ± 0.35
dodecanoic acid	14.8 ± 3.6	14.8 ± 3.74	3.36 ± 0.79	35.54 ± 1.96	18.5 ± 2.26	6.45 ± 3.32	3.0 ± 0.26

<sup>a</sup> Total acid: 97.3% of cheese aroma compound. <sup>b</sup> UT, Ultra-Turrax emulsification. <sup>c</sup> US, ultrasound emulsification.

**Figure 2.** Maltodextrin (50%) stabilized emulsion obtained by ultrasound treatment (power, 140–150 W; sonication time, 10 min) (×40).

quantitative changes, measured by gas chromatography, in the proportions of each volatile. Losses of volatile butyric acid were observed with the modified starch or with the maltodextrin powder obtained after emulsification by the Ultra-Turrax but not with the other experiments. In contrast, an unbalanced aroma in favor of butyric acid was found with modified starch and the ultrasonic method and with the maltodextrin-modified starch for the two emulsification methods. Alone, the powder obtained with the ultrasonic method and maltodextrin as support possessed the same aroma profile as the initial formulation. Amounts of lauric acid, the second most abundant acid, varied in contrary senses: when the butyric acid percentage increased, the lauric acid percentage decreased.

**Surface Aroma and Encapsulation Efficiency.** The amount of aroma compounds absorbed on the surface must be weak to prevent evaporation and/or oxidation. The amount of aroma surface seemed not to be affected by the method of emulsification. The weakest amount was observed with the modified starch support followed by the maltodextrin support and the maltodextrin-modified starch support (Table 4). Surface oil retention is related to the emulsion droplet size (Risch and Reineccius, 1988); the largest microencapsulated particles would be expected to contain less surface oil due to the smaller surface total area. Surprisingly, the

**Table 4. Effect of Emulsification Method and Nature of the Support on the Encapsulation Efficiency**

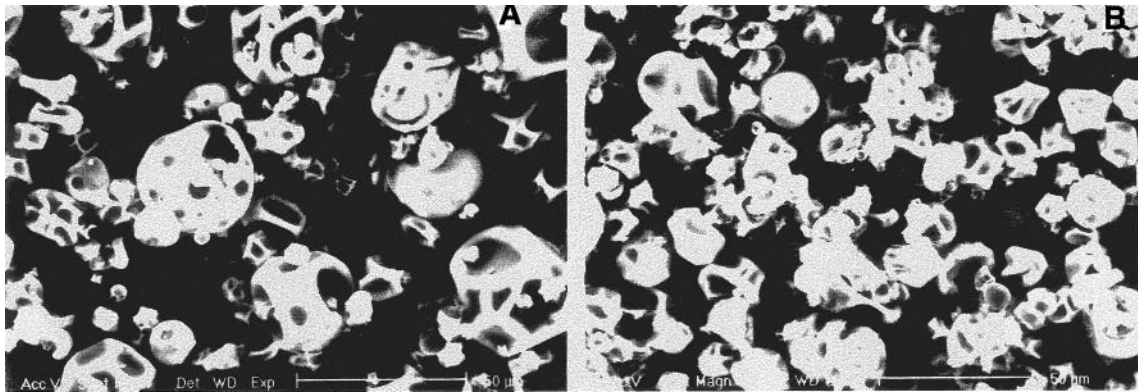
support	emulsification method	aroma surface (mg/100 g of dry wt)	encapsulation efficiency <sup>d</sup> (%)
octenyl succinate starch (40%) <sup>a</sup>	ultrasound <sup>b</sup>	149.7	94.3
	Ultra-Turrax <sup>c</sup>	189.7	83.3
maltodextrin (50%) <sup>a</sup>	ultrasound <sup>b</sup>	289.6	69.8
	Ultra-Turrax	207.6	59.0
maltodextrin/octenyl succinate starch (55%) <sup>a</sup>	ultrasound <sup>b</sup>	779.9	77.7
	Ultra-Turrax <sup>c</sup>	357.3	77.9

<sup>a</sup> Percent of support. <sup>b</sup> Power = 140–150 W. <sup>c</sup> 20500 t/min. <sup>d</sup> Computed on the basis of a theoretical cheese aroma of 20% of the solids (i.e., 17.77 g of butyric acid equivalent/100 g of the solids). EE = [(total fatty acids – surface aroma)/theoretical cheese aroma] × 100.

capsules obtained with maltodextrin-modified starch support, which had the second smallest mean droplet size of 2.2 μm and the second total cheese aroma, exhibited the highest surface oil content. This result shows that factors other than droplet size are controlling the surface oil content. Similar results were found with spray-dried orange oil particles microencapsulated with gum arabic or food proteins (Kim and Morr, 1996; Kim et al., 1996).

The encapsulation efficiency varied from 59% with maltodextrin and Ultra-Turrax to 94.3% with modified starch and ultrasonic treatment (Table 4). Using maltodextrin-modified starch as support, the encapsulation efficiency is equivalent irrespective of the emulsifying method. These results reflect the recovery based on the composition of the original cheese aroma and suppose that microencapsulation resulted in no changes of profile aroma, whereas contrary results have been obtained (see below). It would be expected that the encapsulation efficiencies should be lower when the butyric acid is weakly retained rather than when it is highly retained.

**Microstructural Properties of Spray-Dried Microencapsulated Particles by SEM.** SEM results have shown identical microstructure in shape and surface features for microparticles obtained by ultrasonic or Ultra-Turrax emulsification methods for each support. Modified starch encapsulated particles exhibited highly dented surfaces that were likely due to rapid



**Figure 3.** Scanning electron micrograph of a spray-dried powder prepared with modified starch as support: (A) Ultra-Turrax treatment ( $\times 500$ ); (B) ultrasound treatment ( $\times 500$ ). The figure is reproduced here at 75% of its original size.

shrinkage of the liquid droplets during the early stage of the drying process, whereas maltodextrin-encapsulated particles exhibited a more spherical shape and smoother surface. The coalescence of small particles around large particles resulted in the formation of agglomerates, which have been observed in all powder samples. However, a tangible difference was found in the size of microencapsulated particles due to the homogenization process; with ultrasonic treatment and modified-starch support, the particle size was smaller than with Ultra-Turrax treatment (Figure 3).

#### DISCUSSION

It seems that the efficiency of emulsification is largely determined by the type of wall used as an emulsifier irrespective of the emulsifying method. As previously reported, the use of modified starch results in a better emulsion quality and stability than maltodextrin. It is known that maltodextrins have poor emulsifying properties and emulsion stability but exhibit lower viscosity than modified starch and a good solubility in a high solid range (Reineccius, 1991). The blending of maltodextrin with a highly effective emulsifier such as a modified starch support results in a smaller droplet size. Similar results have been obtained with different starches (Kenyon, 1995) or whey proteins (Young et al., 1993; Sheu and Rosenberg, 1995).

On the other hand, our results have clearly shown that the use of ultrasound increases emulsion quality when the support has low emulsifying properties and a weak viscosity, such as maltodextrin. As previously described (Higgins and Skauen, 1972; Reddy and Fogler, 1980; Eberth and Merry, 1983), the quality of the emulsion depends on an optimum of energy, that is, a power level and an sonication time. Indeed, the acoustic emulsion is formed as a consequence of two phenomena: first, production of large drops by the interfacial instability and, second, breakage into smaller drops by acoustic cavitation. The quality of the maltodextrin emulsion obtained by the action of ultrasound results in a higher aroma retention than the use of Ultra-Turrax and permits limited diffusion of the most volatile and polar compound, that is, butyric acid, during drying.

An increase of aroma retention was also observed using modified starch as the carrier and ultrasonic treatment, whereas no significant difference in droplet size had been shown between the emulsions obtained by Ultra-Turrax and ultrasound. However, other studies with most specific analyses of emulsion ultrastructure have shown that emulsions stabilized by protein isolates

and obtained with an ultrasonic apparatus counteracted the trend to aggregation and gave a smaller droplet size and a broader spectrum of globule size than valve homogenized emulsions (Tornberg, 1980; Krause and Bucheim, 1994). The difference in microcapsule size between the powders obtained by Ultra-Turrax and ultrasound emulsification and observed by SEM is in agreement with the differences found in the aroma retention and in the profiles aroma, that is, the weak retention of butyric acid when Ultra-Turrax is used and the high retention of butyric acid when ultrasound is used. On the other hand, with lemon aroma (data not shown) as core material, no significant difference is found between the retention level of the powders obtained with the modified starch as support and the two emulsification methods. It was observed that the retention rates varies according to the chemical nature of the aroma (Goubet et al., 1998).

Each support material had its own optimum concentration to provide high volatile retention, but generally with a high solid content, the time to form the semi-permeable membrane during drying is shorter than with a weak solid content (Bangs and Reineccius, 1988). Using the maltodextrin-modified starch support, the optimum concentration seems be raised at the highest important total solid used (55%) and explains the retention's not being affected by the emulsification method. On the other hand, the emulsion obtained with ultrasound possessed a higher viscosity (75 cP) than the emulsion obtained by Ultra-Turrax (56 cP). It has been shown that a perceptible increase in viscosity of beverage flavor emulsions is obtained when flocculation, that is, formation of aggregates without coalescence, occurs (Tan and Wu Holmes, 1988). High viscosity led to the formation of large droplets and modified the thickness of the wall, which adversely decreases the drying rate and so the retention level (Risch and Reineccius, 1988; Rosenberg et al., 1990).

The influence of process on the retention of volatile compounds during drying has been extensively studied (Rosenberg et al., 1990; Bhandari et al., 1992). The losses of volatiles occur mainly at stages prior to the formation of dry crust around the drying particle and result in an unbalanced aroma profile. For all samples, the use of ultrasound treatment for emulsification resulted in the strong retention of butyric acid, the most polar and volatile compound, whereas a weak retention is observed for the less polar and volatile compound, lauric acid. These results contradict other studies which have shown that the retention by carbohydrates of high

molecular weight, apolar, and less volatile compounds is favored (Reineccius, 1995; Goubet et al., 1998). Using the maltodextrin-modified starch supports, a strong retention of butyric acid is observed with the two emulsification methods. This specific retention seemed to be independent of methods supposing, as previously stated, that the effect of support was more important than that of emulsification method. Indeed, the predominance of butyric acid in the different powders could be explained by the relative volatility of the compound, by the diffusivity, and by the interactions with the carrier (Goubet et al., 1998). It has been shown that the relative volatility of a compound is affected by the presence of a carbohydrate matrix; the effect depends on the interactions between the particular volatile molecule and the particular carbohydrate. As a general rule, the decrease of the volatility of the compounds was the result of unspecific molecular interactions (Solms et al., 1973; Langourieux and Crouzet, 1994; Godshall, 1997). In the case of starch, the interactions with volatile flavor molecules is understood to be molecular inclusion, a stronger form of interaction (Osman-Ismail and Solms, 1973; Nuessli et al., 1995). The formation of starch inclusion complexes with flavor substances is due to the amylose helix arrangement, which confers particularly hydrophobic characteristic to the interior surface, whereby the molecules with polar and hydrophobic characteristics are entrapped, probably supported by a combination of dipolar and hydrophobic interactions. The molecular structures and physicochemical properties of the flavor substances are known to affect the formation and composition of inclusion complexes (Rutschmann and Solms, 1990; Nuessli et al., 1995). Hydrophobic compounds are known to form complexes that are less stable because these compounds cannot costabilize their complexes by dipolar interactions. Osmans and Solms (1973) have shown, with aliphatic alcohols of different chain lengths, a decrease of molecule amount in inclusion complexes with increasing molecular weight. The inclusion of small linear molecules such as iodine, fatty acids, and decanal was described similarly: in this case only the carbonyl apolar part of the molecules was included in the helix (Rutschmann and Solms, 1990). These observations could explain the selective retention of butyric acid by the modified starch support when emulsification conditions (method and level of solid) favor inclusion complex formation.

## CONCLUSION

Using ultrasound for the emulsification step of cheese aroma and maltodextrin or modified starch appeared to increase emulsion quality and stability and, so, the fatty acids retention of the spray-dried powders. However, the use of ultrasound resulted in an unbalanced aroma in favor of butyric acid.

The study of aroma profiles has shown that it was necessary to consider each fatty acid to estimate the efficiency of carriers and emulsification methods. The knowledge of activity coefficients of aroma compounds and the comprehension of physicochemical interactions between each compound and each support could prevent changes in aroma profiles during microencapsulation or minimize unbalanced aroma through matrix choice.

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