Differences Between Gum Acacias for the Spray Drying of Citrus Oils

By S. J. Risch* and G. A. Reineccius, University of Minnesota, St. Paul, Minnesota

Substantial work has been published which has demonstrated that the flavor encapsulation matrix influences both flavor retention and oxidative stability.[1-5] Several of these studies have included gum acacia as an encapsulation matrix.

Gum acacia, however, was treated as a single product when, in fact, numerous different gum acacias are now on the market and available for flavor encapsulation[6]. It was considered of interest to evaluate different gum acacias for the encapsulation of single fold orange peel oil via spray drying. This is the subject of the research report which follows.

Materials and Methods

This study was conducted in two parts. The first part served as a screening study for the second part.

Matrix Composition: The infeed matrices evaluated in this study are presented in Table I. Capsul and N-Lok were obtained from National Starch Corp., (Bridgewater, NJ), Maltrin M-100 from Grain Processing Corp. (Muscatine, IA) and the gum acacias from Colloides Naturels (Bridgewater, NJ). The gum number 28830 is a traditional gum acacia from Senegal while the other gums are proprietary blends of West African gums.

Table I. Infeed Compositions Evalulated for the Encapsulation of Orange Peel Oil

	Composition (%)			
	Water	Gum/ MS[1]	M-100	Flavor
Part 1				
1. Capsul	65	28	—	7
2. Spraygum IRX 28830	65	28	—	7
3. Spraygum IRX 60642	65	28	—	7
4. Emulgum 29,000 (+M-100)	65	10	18	7
5. Spraygum IRX 61320 (+M-100)	65	18	10	7
6. N-Lok		28	—	7
Part 2				
1. Spraygum IRX 28830	65	28	—	7
2. Spraygum IRX 61320	65	28	—	7
3. Emulgum 29,000 (+M-100)	38.6	17.5	31.5	12.4
4. Spraygum IRX 61320 (+M-100)	38.6	17.5	31.5	12.4
5. N-Lok	38.6	49	—	12.4

[1]MS = modified starch product

Infeed Preparation/Spray Drying: Each encapsulating material was dissolved in hot water (75°C) and then allowed to cool to room temperature prior to spray drying. Just prior to drying, flavor was added (1:4, flavor:carrier solids) to the hydrated matrix with vigorous mixing to accomplish a partial dispersion and it was then homogenized (single pass) using a Manton Gaulin single stage homogenizer (2,500 psig). All samples were spray dried using a Niro Utility Spray Dryer using an inlet air temperature of 200°C and an exit air temperature of 100°C.

Analytical Methods: Moisture content was determined by toluene distillation, total oil by Clevenger distillation and extractable oil by Soxhlet extraction (pentane, 4 hrs.). Oxidative stability was monitored by measuring the formation of limonene oxide by gas chromatography when the product was stored at 37°C. The methods used have been detailed in previous publications.[4]

Emulsion stability was measured by first dissolving 0.1% spray dried sample in water. This solution was placed in a 15 ml centrifuge tube and optical density was measured (400 nm) at centrifugation times (500 Xg) of 0, 5, 10, 15 and 30 min.

*S. J. Risch is currently with Golden Valley Microwave Foods, Minneapolis, MN.

 0272-2666/90/0004-5501$04.00/00

Table II. Total Oil (g/100 g powder) of Spray Dried Samples

Sample	Capsul	Spray-gum IRX 28830	Spray-gum IRX 60642	Emul-gum 29000/ M-100	Spray-gum IRX 61320/ M-100	N-Lok
Trial 1	19.9	18.3	17.5	18.4	16.9	20.0
Trial 2	20.0	17.8	17.7	18.5	17.2	20.0
Average	20.0	18.1	17.6	18.5	17.1	20.0

Table III. Extractable Oil (mg/100 g powder)

Sample	Capsul	Spray-gum IRX 28830	Spray-gum IRX 60642	Emul-gum 29000/ M-100	Spray-gum IRX 61320/ M-100	N-Lok
Trial 1	30.6	36.3	39.5	33.9	76.3	15.4
Trial 2	32.6	40.7	39.6	28.8	77.1	4.2(?)
Average	31.6	38.5	39.6	31.4	76.7	15.4

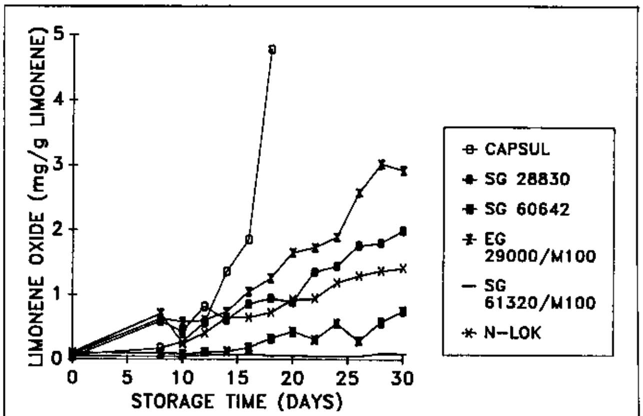

Figure 1. The oxidative deterioration of encapsulated orange oil (as measured by limonene epoxide formation) during storage.

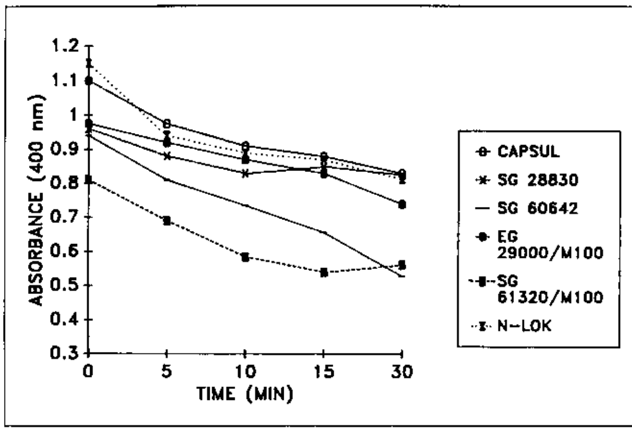

Figure 2. Emulsion stability (as measured by absorbance at 400 nm) of reconstituted spray dried orange oils during centrifugation at 500 times g.

Results and Discussion

PART 1: The results of the total oil analyses are shown in Table II. The two modified starches, Capsul and N-Lok showed 100% retention of the orange oil. The gum arabic and gum arabic/M-100 samples ranged from 86 to 93% retention. One should note that the values presented in Table II are in grams oil/100 g powder (not corrected for moisture). Very commonly the industry chooses to express volatile oil in ml oil/100 g powder. The numbers presented in Table II should be divided by 0.84 to get the volume expression which is more common in the industry. Virtually all of the infeed materials were easily pumped and atomized and thus could have been used at higher solids contents.

The results of the extractable oil analyses are shown in Table III. The amount of extractable oil was essentially the same for the first four samples, with the values ranging from 31.4 to 39.6 mg oil/100 g powder. Spraygum IRX 61320/M-100 blend had the highest amount with 76.7 mg oil/100 g powder while N-Lok had the lowest level with 15.4 mg oil/100 g powder.

The amounts of limonene oxide formed with time when the samples were stored at 37°C are presented graphically in Figure 1. Capsul had the shortest shelf. It lasted 16 days at 37°C before the level of limonene oxide exceeded 2.0 mg/g limonene. This value was arbitrarily chosen as the end of shelf-life for all samples of the encapsulation matrices examined.

Spraygum IRX 61320/M-100 was by far the best, showing essentially no formation of limonene oxide after 30 days at elevated temperature. Spraygum IRX 60642 was also better than any of the remaining samples. This sample had not reached the end of shelf life when the study was terminated at 30 days storage. Of the samples investigated, the spraygum 61320/M-100 combination and SG 60642 exhibited the best shelf-stability.

The results of the emulsion stability test are shown in Figure 2. While one is inclined to compare the initial absorbance readings of the different emulsions, our experience has been that this is not a good means of comparing emulsion quality.

In theory, the higher the absorbance, the smaller the particle size distribution and thus more stable the emulsion. We have found that different emulsifying agents (e.g. gum acacias or modified starches) have different light absorption properties (also a function of light wave length) irrespective of the emulsion particle size distribution.

While we do not have a good method to rapidly

Table IV. Total Oil (g/100 g powder) of Spray Dried Samples

Sample	Spray-gum IRX 61320	.Spray-gum 28830	Emul-gum 29000/ M-100	N-Lok	Spray-gum IRX 61320/ M-100
Trial 1	14.52	18.37	16.66	22.13	15.91
Trial 2	15.22	19.71	15.22	21.10	15.91
Average	14.87	19.04	15.94	21.62	15.91

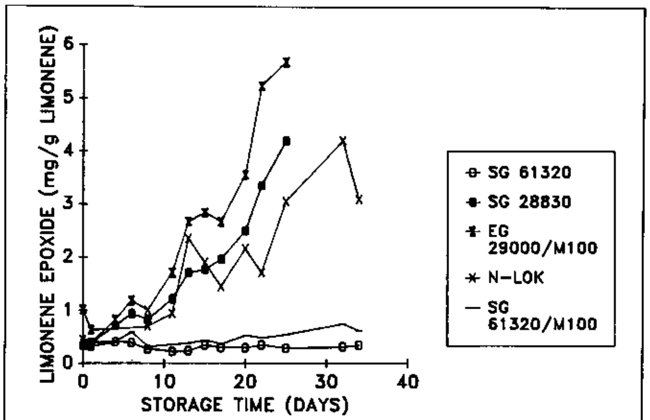

Figure 3. The oxidative deterioration of encapsulated orange oil during storage.

measure emulsion stability, this is the subject of a thesis project within our group. We currently are evaluating emulsion quality by how rapidly it destabilizes during centrifugation (i.e., rate of decline in optical density during centrifugation).

Based on the rate of change in optical density during the first 10 minutes of centrifugation, Capsul and N-Lok would be considered to form the least stable emulsions. The SG 61320/M-100 sample and SG 60642 were next least stable being approximately equivalent. The EG 29000/M-100 was next followed by the pure traditional gum SG 28830.

It is somewhat of interest that the EG 29000/M-100 blend was nearly equivalent to a pure gum acacia (e.g. SG 28830). One would have expected this blend to be substantially less stable than a pure gum acacia system since the M-100 offers no emulsification properties.

PART 2: Based on the results obtained in Part 1, several changes were made in choice of encapsulating agent. The traditional acacia (28830) was included for replication. Since the SG 61320 performed so well as a blend, it was decided to include it as a pure carrier. The two gum/M-100 blends and N-Lok were also included but were used at higher infeed solids levels. For this experiment, data are presented only for the most critical concerns, retention and shelf-life.

The results of the total oil analyses are shown in Table IV. The N-Lok showed better than 100% retention of the orange oil. The better than possible retention was the result of skimming the foam off the sample prior to adding the orange oil. This removed some of the solids but the same amount of orange oil was added. The gum acacias and gum acacia/M-100 samples ranged from 74 to 96% retention of the added oil.

Spraygum IRX 28830 (traditional gum) showed the best retention of the gum acacias examined. It would be expected that increasing the solids level would increase the retention of oil; however, only a slight increase was observed for Spraygum IRX 61320 when M-100 was added to it.

The amounts of limonene oxide formed with time when the samples were stored at 37°C are presented in Figure 3. Spraygum IRX 61320 and Spraygum 61320/M-100 were by far the best, showing essentially no formation of limonene oxide after 30 days at elevated temperature. The limonene oxide values for N-Lok exceeded 2 mg/g limonene after 17 days at 37°C. Spraygum IRX 28830 lasted 17 days at 37°C while Emulgum 29000/M-100 lasted only 11 days at 37°C.

Conclusions

If one looks at the data overall, the modified starches rate excellent in terms of flavor retention and quite acceptable in terms of emulsion stability. The primary weakness of these materials is that they provide very poor protection (Capsul) or moderate protection (N-Lok) against oxidation. If one is drying a flavor containing citrus oils (or perhaps other oxidizable components such as benzaldehyde), antioxidants must be added in order to give the desired shelf-life.

The addition of antioxidants, as well as the modified starches in themselves, preclude an "all natural" label. If label requirements demand an all natural product or the company does not wish to label antioxidants, the modified starches are not good choices for flavor encapsulation.

The gum acacias were found to be quite different in terms of flavor retention and protection against oxidation. The Spraygum IRX 61320 was found to give excellent shelf-life, emulsion stability and satisfactory retention. This product alone or in combination with an inexpensive carrier such as Maltrin M-100 appears to yield a cost effective product with good performance.

Acknowledgements: *Published as paper no. 17,616 of the Minnesota Agricultural Experimentation Station based on research conducted under project 18-83.*

References

Address correspondence to Dr. Gary A. Reineccius, Department of Food Science and Nutrition, University of Minnesota, St. Paul, MN 55108, USA

1. WE Bangs and GA Reineccius, Corn starch derivatives: Possible wall materials for spray-dried flavor manufacture. In: *Flavor Encapsulation.* SJ Risch and GA Reineccius, eds; ACS Symposium Series No. 370, Washington, DC (1988) p. 12
2. GE Inglett, P Gelbman and GA Reineccius, Encapsulation of orange oil. Use of oligosaccharides from alpha amylase modified starches of maize, rice, cassava and potato, *ibid* p. 29
3. MM Leahy, S Anandaraman, WE Bangs and GA Reineccius, Spray drying of food flavors II. A comparison of encapsulating agents for the drying of artificial flavors, *Perfum Flavorist* **8**(5):49 (1983)
4. W Baisier and GA Reineccius, Spray drying of food flavors V. Factors influencing shelf-life of encapsulated orange peel oil. *Perfum Flavorist* **14**:48 (1989)
5. PC Trubiano and NL Lacourse, Emulsion stabilizing starches: use in flavor encapsulation. In: *Flavor Encapsulation,* SJ Risch and GA Reineccius eds, ACS Symposium Series No. 370, Washington, DC, (1988) p. 45
6. F Thevenet, Acacia gums: stabilizers for flavor encapsulation, *ibid* p. 37.