

DEVELOPMENT OF GREEN APPLE FRAGRANCE MICROCAPSULES USING COACERVATION TECHNOLOGY



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Abstract

This study investigates the optimal conditions for encapsulating green apple flavor compounds using the coacervation method. Gum arabic and gelatin were employed as coating materials, with glycerol acting as the cross-linking agent. A preliminary analysis of green apple samples was conducted using gas chromatography-mass spectrometry (GC-MS) to identify the primary flavor compounds. Ethyl valerate was identified as the key compound, with a total ion chromatogram (TIC) peak observed at a retention time of 4.65 minutes. The study evaluated various emulsification rates and coating material concentrations to determine the optimal encapsulation conditions, assessed based on encapsulation efficiency (entrapment efficiency). Emulsification speeds of 18,000 and 24,000 rpm, along with a coating material concentration of 5.0% w/v, were investigated. The resulting microcapsules exhibited a spherical morphology with a single-layer coating. At a coating material concentration of 5.0% w/v, the microcapsule thickness and diameter were measured as $48.89 \pm 8.18 \mu\text{m}$ and $44.59 \pm 8.22 \mu\text{m}$ for emulsification speeds of 18,000 and 24,000 rpm, respectively. The results suggest that a higher coating material concentration leads to increased microcapsule thickness, while higher emulsification speeds reduce capsule size with minimal effect on thickness. In future experiments, the entrapment efficiency (%) will be assessed to evaluate the release profile of flavor compounds from the microcapsules over a two-week period. Additionally, the preservation efficiency of the 5.0% w/v coating material will be examined by measuring the relative surface oil content.

Introduction



Figure 1 Green apple fragrance oil

Green apple fragrance

Green apple fragrance is fresh, crisp, and slightly tangy, with a sweet-tart, fruity, and invigorating scent.

Common Uses : Food industrial
Perfume and body spray
Personal care product
Household product

Scientific name : Ethyl pentanoate

Chemical formula : $\text{C}_7\text{H}_{14}\text{O}_2$

Molecule weight : 130.18 g/mol

Boiling point : 144-145°C

Physical appearance: Clear, colorless, or pale yellow liquid.

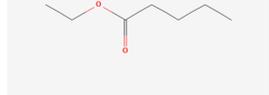


Figure 2 Structure of Ethyl Valerate



Figure 3 Microcapsules

Encapsulation

- Encapsulation refers to a process used to contain and protect fragrance molecules within microcapsules.
- Encapsulation technology allows fragrance to be released gradually under specific conditions, ensuring a longer-lasting scent.

Coacervation

- The microencapsulation process using coacervation forms a protective polymer shell around fragrance oil.
- The final microcapsule, made of gelatin and gum arabic, slows evaporation and enables controlled fragrance release through heat or friction.

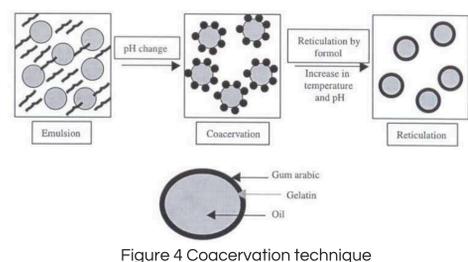


Figure 4 Coacervation technique

Results

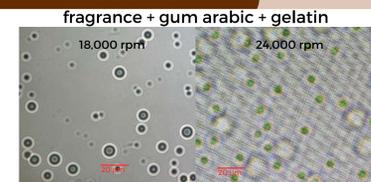


Figure 5 Microcapsules in different rpm

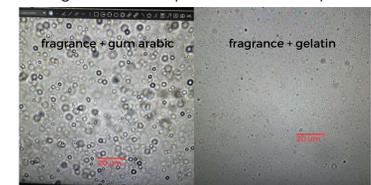


Figure 6 Microcapsules in different condition



Figure 7 microcapsules after undergoing the freeze-drying process.

Microcapsules

The microcapsule thickness and diameter were measured as $48.89 \pm 8.18 \mu\text{m}$ and $44.59 \pm 8.22 \mu\text{m}$ for emulsification speeds of 18,000 and 24,000 rpm

Compared to fragrance + gelatin, fragrance + gum arabic showed more dispersed fragrance oil droplets and well-defined spherical microcapsules, while gelatin forms smaller and less spherical microcapsules.

Determination of Ethyl valerate

The peak at $m/z = 88$ is the characteristic and abundant ion for Ethyl valerate.

$t_R = 4.65 \text{ min.}$

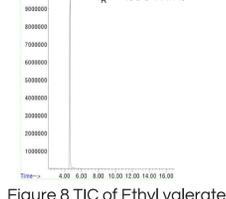


Figure 8 TIC of Ethyl valerate

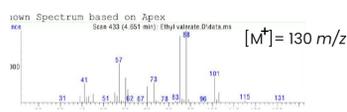
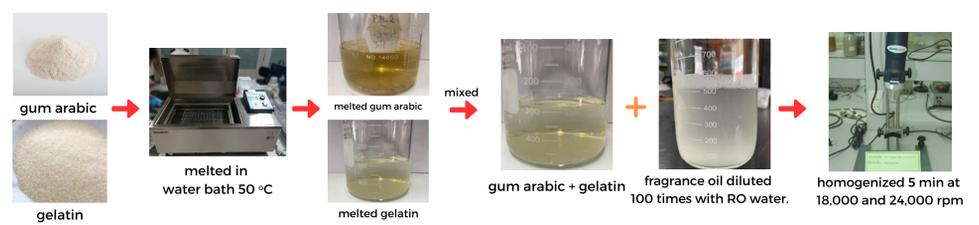
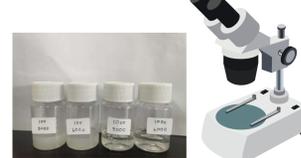


Figure 9 Mass spectrum of Ethyl valerate

Material & Method



Microcapsules under a microscope.



collected 0.5 mL of emulsion for microscopy.

Adjusted pH



emulsion pH 6



adjusted pH to 4



adjusted pH to 6

Freeze dried



microcapsules



freeze dryer



freezed -80°C



microcapsules after adjusted pH

Future analysis

Surface oil

DCM extraction
Liquid phase → evaporated

Total oil

DCM extraction
Liquid phase
Solvent extraction
Evaporation

GC-MS analysis

GC-MS condition % Entrapment Efficient % Entrapment Efficiency = $\frac{\text{Total oil} - \text{Surface oil}}{\text{Total oil}} \times 100\%$

Stationary phase column: HP-5, 30 meters in length, 0.25 mm in diameter, with a stationary phase thickness of 0.25 microns.
Initial temperature: 80°C (held for 6 minutes), then increased at a rate of 3°C per minute until reaching 250°C.
Auxiliary temperature: 250°C.
Quadrupole temperature: 150°C
MS scan mass range: 29-550 m/z
Injection volume: 1 microliter.
Carrier gas: Helium, with a flow rate of 1 mL/min.
Scan speed: 1000 amu/s

Results

- The microencapsulation process could create spherical microcapsules with a core and a wall material. Stirring speed affects the size of the microcapsules:
High speed (24,000 rpm) → Smaller microcapsules
Low speed (18,000 rpm) → Larger microcapsules
- The microcapsule thickness and diameter were measured as $48.89 \pm 8.18 \mu\text{m}$ and $44.59 \pm 8.22 \mu\text{m}$ for emulsification speeds of 18,000 and 24,000 rpm
- The physical characteristics after the freeze-drying process changed from a liquid or gel state to a solid that was chewy and dry, while the fragrance remained after the processed.
- GC-MS results showed the presence of ethyl valerate at 4.65 minutes

Futurework

- The entrapment efficiency (%) will be assessed to evaluate the release profile of flavor compounds from the microcapsules over a two-week period.
- The preservation efficiency of the 5.0% w/v coating material will be examined by measuring the relative surface oil content.

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