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Microcapsule Formation by Coacervation

Practical 1 - Bench formulation practical

Aim

To compare phase-separation conditions during microcapsule formation.

Principle

Coacervation or phase separation produces a polymer-rich phase that may deposit around a dispersed core. Temperature change, non-solvent addition and an incompatible polymer can alter polymer solvation and separation.

Formation of a polymer-rich phase does not by itself establish a complete capsule wall; morphology and core retention must be evaluated.

A formulation comparison is meaningful when the raw-material grades, batch size and evaluation conditions are recorded. Change one planned factor at a time or use a documented experimental design, and compare independently prepared batches rather than only repeated readings from one batch.

Materials and Requirements

- Approved core material and coating polymer
- Selected solvent and non-solvent system
- Temperature-controlled mixing and microscopy
- Calibrated balance, suitable containers and identified evaluation equipment
- Controlled batch sheet and raw-data forms

Procedure

1. Define the purpose, formula, batch size, material grades and comparison conditions. Calculate all quantities before weighing.
2. Check equipment readiness and record material identities and actual quantities. Reconcile theoretical and actual batch mass.
3. Select the prescribed baseline formulation and keep core loading and mixing conditions fixed across the comparison.
4. Apply one approved phase-separation trigger at a time: temperature change, non-solvent addition or incompatible-polymer addition.
5. Record the addition order, conditions and onset of phase separation using microscopy or other suitable observations.
6. Recover and stabilise the preparation through the adopted process, then assess morphology, size, core retention and release.
7. Evaluate the stated quality attributes on representative portions using the same method for all comparison batches. Record individual readings and any excluded observations with reasons.
8. Calculate the specified response, variability and material balance. Compare with predefined teaching or product criteria and discuss sources of variation.
9. Label retained preparations and dispose of remaining material through the laboratory procedure. Keep the complete preparation and evaluation record together.

Observations and Record

Enter actual measurements or the supplied teaching data. Record the method, material identity, conditions and units.

Attribute or stage	Method / conditions	Individual readings	Calculation / comment
Trigger and conditions	Record the adopted method	Record	Evaluate
Phase-separation observation	Record the adopted method	Record	Evaluate
Size / morphology	Record the adopted method	Record	Evaluate
Core retention	Record the adopted method	Record	Evaluate

Calculations

• Core retention (%) = measured retained core / input core x 100
• Dry recovery (%) = recovered dry mass / theoretical input solids x 100

Worked Example

Illustrative dry input solids 2.00 g and recovered material 1.60 g give 80.0% dry recovery.

Measured retained core 0.36 g from 0.50 g input gives 72.0% core retention.

Report morphology as well as these mass-based values; a precipitate may have high recovery without having the intended microcapsule structure.

Result and Interpretation

• Compare trigger conditions and morphology using predefined descriptors. Explain the distinction between precipitation, microsphere formation and encapsulation.
• State the preparation conditions and the evidence supporting the result. A satisfactory physical observation alone does not establish long-term stability, clinical performance or suitability for administration.

Precautions

• Use the approved solvent system and ventilation.
• Avoid changing multiple phase-separation factors unintentionally.
• Use the specified stabilisation process rather than introducing an unqualified reactive cross-linker.
• Use the laboratory-approved formulation and equipment procedure. Teaching batches are identified for laboratory evaluation.

Viva Questions and Answers

What is a coacervate?

A polymer-rich liquid phase formed under suitable phase-separation conditions.

Why examine morphology?

Mass recovery alone does not prove the intended capsule structure.

Why control mixing?

It affects dispersion and the contact between phases.

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