Experiment 51

Formulation Analysis: Design-Expert Software

Objective: To analyze and optimize pharmaceutical formulation data using Design-Expert software by:

1. Modeling factor–response relationship.
2. Identifying critical formulation parameters.
3. Determining optimal factor levels to achieve target product quality.

THEORY

Design-Expert is a widely used software for **Design of Experimen***ts (DoE)*. It allows formulation scientists to:

- Build factorial and response surface designs.
- Evaluate main effects and interactions.
- Predict responses and optimize formulation parameters.

In pharmaceutical development, this reduces trial-and-error, improves product quality, and identifies robust factor ranges.

MATERIALS AND SOFTWARE

- Computer with **Design-Expert software** installed v12 or latest version
- Formulation experimental dataset (Excel or CSV)
- Spreadsheet software (Excel) for data preparation
- Plotting software (optional, for customized graphs)

EXPERIMENTAL DESIGN OVERVIEW

Step 1: Define Factors and Responses

- **Factors (Independent Variables):** For example, polymer %, binder %, compression force
- **Responses (Dependent Variables/CQAs):** For example, tablet hardness, dissolution %, friability, assay

Step 2: Select Design Type

- Factorial (full or fractional) for screening
- Response Surface Designs (Central Composite Design or Box-Behnken) for optimization

Step 3: Generate Experimental Matrix

- Randomized runs using software
- Include replicates and center points for error estimation

PROCEDURE

Step A: Create Project in Design-Expert

1. Open Design-Expert and select **New Design**.
2. Choose the design type: Factorial, CCD, or Box-Behnken.
3. Enter the **number of factors** and levels (e.g. 3 factors at 3 levels).
4. Define **response variables** and their units.

Step B: Enter Data

1. If running new experiments: Fill in **factor levels** for each run.
2. If using pre-collected data: Import data from Excel/CSV.
3. Verify all data entries; ensure units are consistent.

Step C: Fit Model

1. Select model type: Linear, 2FI (two-factor interaction), Quadratic, or Special Cubic.
2. Click **Analyze** to fit the model.
3. Check model summary: R^2, Adjusted R^2, Predicted R^2, Adequate Precision.
4. Review ANOVA table to identify statistically significant factors ($p < 0.05$).

Step D: Examine Factor Effects

1. Generate **Main Effects Plots** for each factor.
2. Generate **Interaction Plots** to see factor–factor interaction.
3. Use **Contour Plots** and **3D Surface Plots** to visualize relationship between factors and responses.

Step E: Optimization

1. Open **Optimization Tab** in Design-Expert.
2. Set **desired goals** for each response: Maximize, minimize, target value.
3. Generate **desirability function** to find optimal factor settings.
4. Review predicted responses and overlay plots to validate solution.

Step F: Validation (Optional)

1. Run experiment at predicted optimal settings.
2. Compare observed *vs* predicted responses to check model accuracy.

DATA RECORDING

Example Table for Input Data (Factors and Responses)

Run	*Polymer % (A)*	*Polymer % (B)*	*Compression force (kN) (C)*	*Hardness (kg)*	*Dissolution %*	*Friability %*
1	10	5	5			
2	10	5	15			
...	...	...	...			

Example Table for Optimized Formulation

Factor	*Optimal level*	*Predicted response*	*Observed response*
A (%)	15	85	84
B (%)	10	95	96
C (kN)	12	3	3.2

RESULTS AND INTERPRETATION

- Identify **critical factors** affecting each CQA.
- Examine **interaction effects** between factors.
- Use overlay and desirability plots to propose **robust optimal formulation**.
- Compare predicted and observed responses to validate model.

CONCLUSION