UNIVERSITÀ
DEGLI STUDI DI MILANO-BICOCCA

COURSE SYLLABUS

Analytical Methods in Formulation Chemistry

2122-2-F5401Q060

Aims

The main objective of the course is to provide the student with the theoretical foundations and operational tools of the main analytical strategies for the rational optimization of formulations and for the adequate treatment of the information contained in the experimental chemical data, so as to allow the development of a science-based approach that focuses the mechanism of action of each component that contributes to the achievement of the required functionality of the formulation. Knowledge of the principles and operating conditions of the main techniques in the field of experimental design for formulations and mixtures will be developed together with the ability to choose and manage the most suitable investigation approaches for the purposes of the analysis. The student will then be able to evaluate the characteristics of the fundamental approaches, the fields of application, to identify their advantages and disadvantages and will therefore be able to suggest the choice of the solution deemed most suitable for a specific problem.

In particular, at the end of the course, the student must demonstrate that he/she is able to achieve the following formative objectives:

Knowledge and understanding:

- describe the main analytical strategies for the formulations presented in the course
- describe the fundamental parameters for the evaluation of the results
- describe the advantages and disadvantages of the different approaches

Applying knowledge and understanding

- select the most suitable approach to deal with a specific problem
- evaluate alternative methods to face the problem

o concretely apply the selected methods and calculate the related statistical information

Making judgements

o acquire knowledge and skills to develop a critical understanding of the main analytical strategies for the formulations

o justify a critical discussion on the methods used and the information obtained

Learning ability

o understand the different approaches and their methodological application in order to use them correctly when analysis a specific formulation problem

Contents

Introduction to experimental design. Experimental designs for screening. Regression models for experimental design. Experimental design for optimization. Experimental domain for mixtures. Modelling and designs for the study of formulations. Practical experiences in the laboratory to acquire the tools and methods of analysis.

Detailed program

Introduction to Experimental designs for mixtures. Introduction to experimental design: terminology and methodology of experimental research: analysis of the problem, planning of experiments, execution of the experiments, data analysis and modelling. Full factorial designs for factor screening: factor coding, experimental plan and experimental matrix, calculation of effects and their variance, calculation of coefficients with the rule of signs. Fractional factorial designs: confounded effects and resolution. Plackett-Burman designs and use of dummy factors. Regression models for experimental design, coefficient estimation, information and dispersion matrices, variance of residuals as an estimate of experimental variance, degrees of freedom in experimental design. Regression model diagnostics: ANOVA and lack of fit, residuals, normal probability plots, precision of predictions, confidence intervals for coefficients, validation trough test points. Displacement and optimisation: the simplex and path ascent method, three-level full factorial designs, the central composite designs, Dohelert's designs, Box-Behnken's designs, D-optimal designs. Response surfaces. Multi-response modeling: desirability and the Pareto front. Examples of applications of experimental design for independent factors.

Experiment designs for formulations. Experimental domain for mixtures and simplex, ternary plots. Regression methods for the designs of mixtures: canonical polynomials, interpretation of coefficients and effects. Designs for mixtures: Simplex-Lattice and Simplex-Centroid Designs. Regular and irregular experimental domains for mixtures: lower and upper limits, consistency of limits, pseudo components, determination of simplex domain and number of vertices. Experimental designs for mixtures with regular and irregular bound domains. Candidates points for irregular domains. D-optimal and checkpoint identification for mixtures. Strategies for the selection of significant components to optimize the functionality of the formulation. Approaches for the study of systems including mixtures and independent factors. Examples of applications of experimental design for the study of formulations.

Practical experiences in the laboratory to acquire the tools and methods of analysis.

Prerequisites

Basic knowledge on the main elementary statistical indices and multivariate analysis, concept of experimental variance, basic computer operating skills in practical laboratory experiences.

Teaching form

The course is divided into a part of lectures, where the theoretical and practical notions are provided. Practical laboratory experiences are also provided in order to acquire the tools and operating procedures of the analytical methods for the chemistry of the formulations.

Textbook and teaching resource

The slides of the lessons are provided on the e-learning page of the course. In addition, the teachers provide via e-learning platform an electronic book on the fundamentals of experimental design. In addition to the material provided by the teacher, the following textbook may be useful: Gareth A. Lewis, Didier Mathieu, Roger Phan-Tan-Luu, Pharmaceutical experimental design, Dekker, New York, 1999. The book is available in electronic format in the university library. Finally, Excel and Matlab tools are provided for the practical laboratory sessions.

Semester

Second semester

Assessment method

The exam consists of an oral examination, where topics presented in the lessons are discussed. In the oral exam a report on a case study provided by the teacher and developed by the student is also discussed. In addition to the theoretical fundamentals given in the course, students' skills and aptitudes are also assessed to adapt the theoretical foundations to particular operative and practical conditions; the expositive ability and adequacy of the student's language is also assessed. The exam can be taken on request in English.

Office hours

Teachers receive students in their offices upon an e-mail request.