Chemical Product Engineering and Design: Active Learning through the Use of Case Studies

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Abstract – **This paper presents a recently developed web resource of sample problems to teach chemical product design, which is still a not very well established discipline within chemical engineering. The website is organised according to a general template for product development and incorporates some recently proposed concepts to systematise the discipline of chemical product engineering. A case study of a cosmetic lotion is presented in more detail, illustrating the benefits of inductive learning as well as specific topics, namely integrated product/process design and the relationship between product composition and structure and product performance.**

Index Terms – Chemical product design, Chemical product engineering, Emulsions, Learning through case studies.

INTRODUCTION

New product development, which involves the integration of strategic and management actions with technical efforts, is a crucial activity to the success of any corporation.

Whilst in some industrial and engineering sectors, such as the mechanical and electronic, the technical side of product development has always been heavily emphasised [1], in the chemical process industries the systematic and efficient design of new products is a relatively recent concern. Traditionally, the design of chemical products, such as a hair conditioner, has been led as an empirical art relying on companies' internal background, trade secrets and trial-and-error formulation procedures. However, this approach has become incompatible with the current dynamic and demanding markets, and the concepts of chemical product engineering and chemical product design have been emerging within chemical engineering science and practice [2-5].

One of the major challenges in the teaching of chemical product design is to find and develop plausible and illustrative examples, in part because industrial innovation practices are largely undisclosed. The Engineering Subject Centre (UK) has recently sponsored a project aiming at producing a website with examples for chemical product design teaching, which will be presented in this paper. A particular example of a cosmetic product will be explored in more detail.

CHEMICAL PRODUCT DESIGN WEBSITE

The core of the website is organised in six sections (Table I), each of them corresponding to a different stage of product development [1]. Each section presents an overview of approaches and tools relevant for the corresponding design stage and one or more case studies illustrating the kind of problems addressed in that particular stage. The case studies were developed to motivate students to deal with marketing and management topics, which are typically not incorporated in their curricula, as well as show them how chemical engineering core concepts (for example thermodynamics and transfer phenomena) can be applied to design a new chemical product. In addition, they illustrate a wide variety of chemical products including formulated consumer products (paints, beverages, perfumes and cosmetics), functional materials (fiber-reinforced polymers) and physico-chemical-based devices (cooling system for drinks).

TABLE I
CORE SECTIONS OF THE WEBSITE AND CORRESPONDENT CASE STUDIES.

Section	Case study
Identify customer needs	Developing a new concept of paint
Set target performance specifications	Comfort in beverages: a portable cooling system for drinks
Generate product ideas	A long lasting fizzy drink
Select product ideas	1. Developing a UV barrier film 2. Computer-aided design of a fibre-reinforced polymer composite for application in a storage tank
Set final product specifications	Engineering a perfume
Process design	Integrated product and process design for a hydrating body lotion

In the introductory section of the website, a conceptual model for the chemical product engineering discipline [2] is briefly discussed. Chemical product engineering is understood as the broad area of knowledge that supports the operational task of of chemical product design, including product-related scientific topics that should be reinforced in the chemical engineering curricula, such as molecular modelling, colloid chemistry, rheology and dispersed systems. One of the basic pillars of chemical product engineering is the development of property, process and usage functions, relating technical specifications (product composition, structure, processing and usage conditions) to product functionalities, also designated as quality factors. These relationships may be represented by the chemical product pyramid of Figure 1.

The site also includes a section with references for further information and reading, gathered by subject and that can also be quickly accessed by clicking the respective numbers throughout the website. Finally, there is a section of experience sharing, where new sample problems can be proposed and teaching and professional experiences exchanged.

For further details and a more vivid experience of chemical product design concepts, tools and examples, please visit the website, available since April 2007, on http://www.engsc.ac.uk/an/mini_projects/cpd/index.html.

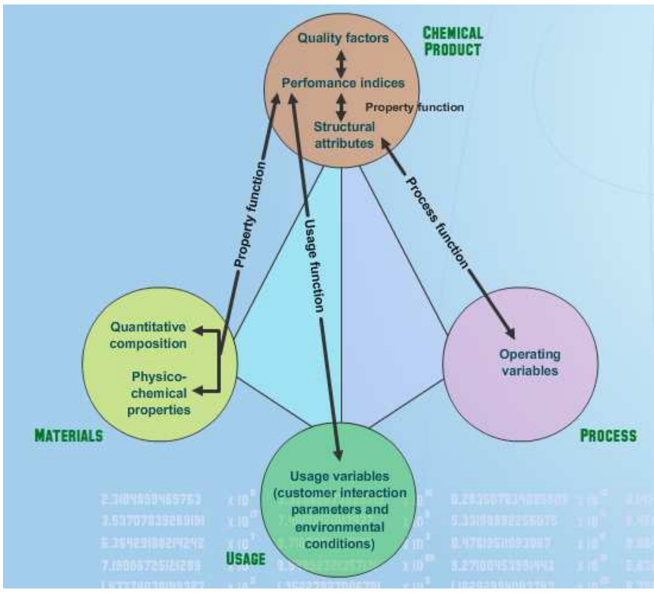

FIGURE 1
CHEMICAL PRODUCT PYRAMID.

COSMETIC LOTION CASE STUDY

The case study here described in more detail exemplifies the design of the manufacturing process for a formulated product and also how product and process design decisions may interact. Property, process and usage functions are developed and used to derive an integrated product/process optimal solution.

The main function of the body lotion under study is to maintain the skin moisturised by supplying water to skin and reducing the water loss from it. Other important quality factors are stability, flow properties and several sensorial attributes [6]. The product development process has reached a stage at which the formulation shown in Table II has been specified for the new lotion. It is an oil-in-water emulsion, with the oil-phase containing occlusive agents and the aqueous phase a humectant (glycerol) and a thickener (xanthan gum, a polysaccharide). The emulsion formation and stability are promoted by two emulsifiers whose molecules adsorb at the surface of oil droplets. A small amount of preservatives and fragrances is also included in the formulation. The product design variables not yet specified are the mass percentages of xanthan gum (w_T) and glycerol (w_G) in the aqueous phase and the oil-phase volume percentage (ϕ). These variables have to be set under the limits shown in Table II.

The so far specified formulation guarantees most of the product functionalities, and at this stage only two quality factors are addressed: the so-called skin feeling, which refers to the sensations experienced during lotion application on skin, and the smoothness. Sensory panel tests reveal that skin feeling is correlated with product viscosity and it is known that smoothness is related to the size of oil droplets. Viscosity depends on the product design variables not yet specified and droplet size is in part controlled by the mixing conditions in the lotion manufacturing process. These dependences have to be quantified and the product/process design problem then addressed in an integrated way. This is accomplished in four steps as follows.

Step 1. Develop quality models for the product

Sensory panel tests [7], discriminated in sensations at the beginning and end of lotion application on skin, indicate a significant correlation between these two quality factors and the lotion viscosity at the correspondent shear rates of application, μ_1 and μ_2 respectively (performance indices).

TABLE II
BODY LOTION FORMULATION.

Part A (10 to 15% (v/v) of the total)	% (w/w)	Part B	% (w/w)
Stearic acid (occlusive)	25.5	Deionized water (solvent)	q.s.
Cetyl alcohol (occlusive)	10.3	Glycerol (humectant)	5-12
Petrolatum USP (occlusive)	10.3	Xanthan gum (thickener)	0.5-1.5
Mineral oil, 70 mPa.s (occlusive)	20.5		100
Isopropyl palmitate (occlusive)	20.5	Part C (~0.25%(w/w) of the total)	
Glyceryl monostearate (emulsifier)	10.3	Preservatives	
PEG-40 stearate (emulsifier)	2.6	Fragrances	
	100		

From such results, Taguchi loss functions of the form $L = k(\mu - \mu^*)^2$ are derived for the initial and final skin feeling, where L is the product lost value (in %) associated with a deviation of the viscosity μ from its ideal value μ^*, and k is a loss coefficient. The ideal values are $\mu_1^* = 374.7$ Pa.s and $\mu_2^* = 0.0242$ Pa.s.

Regarding smoothness and its relation with droplet size, the data are scarce and uncertain and hence a simpler approach is taken, with the product being considered satisfactory once $D_{max} \leq 10$ μm, where D_{max} is the maximum droplet diameter.

Step 2. Develop property and usage functions

Experimental data are available for the viscosity of aqueous solutions of xanthan gum [8] (Figure 2), showing that three different regions may be distinguished: for lower values of shear rate (γ), the behaviour is approximately Newtonian; intermediate values of shear rate correspond to a strong shear-thinning region; for high values of shear rate, the behaviour can be considered Newtonian again. These data are fitted to a Carreau model, in which the glycerol contribution is incorporated assuming that it only influences the limiting viscosity for high shear rates. One thus obtains a model for the viscosity of the lotion continuous phase: $\mu_c = \mu_c(w_T, w_G, \gamma)$. The theoretical model of Yaron and Gal-Or [9] is then used to predict emulsion viscosity from single-phase individual viscosities: $\mu = \mu(\mu_c, \mu_d, \phi)$, where μ_d stands for the oil-phase (Newtonian) viscosity equals to 0.0654 Pa.s. The complete model for the viscosity of the body lotion has then the form: $\mu = \mu(w_T, w_G, \phi, \gamma)$. The dependence of the lotion viscosity μ on the shear rate γ is similar to that of aqueous solutions of xanthan gum, shown in Figure 2.

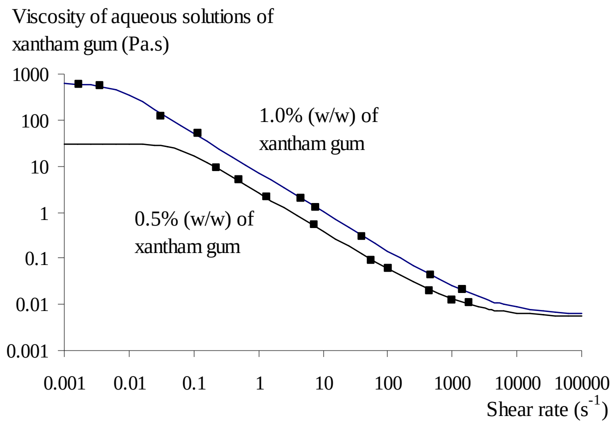

FIGURE 2
VISCOSITY OF AQUEOUS SOLUTIONS OF XANTHAM GUM (EXPERIMENTAL DATA AND FITTED MODEL).

The model above can be used to predict the two performance indices μ_1 and μ_2 once the correspondent shear rates of lotion application are known:

$$\mu_1 = \mu_1\left(w_T, w_G, \phi, \gamma_1\right) \tag{1}$$

$$\mu_2 = \mu_2\left(w_T, w_G, \phi, \gamma_2\right) \tag{2}$$

The initial viscosity is that perceived at the beginning of lotion application on skin, when the lotion starts to flow readily. This corresponds to the transition point between the Newtonian region for low shear rates and the strong shear thinning-region, calculated as the inflexion point of the curve $\mu(\gamma)$. At the end of lotion application, the lotion is spread rapidly over a large skin area with a correspondent large shear rate, whose typical value is $\gamma_2 = 5000$ s^{-1} [7].

Equations (1) and (2) relate the performance indices μ_1 and μ_2 to the composition of the lotion (in particular, w_T, w_G and ϕ), and therefore, in the language of the chemical product pyramid (Figure 1), they are property functions. But these equations also incorporate the dependence of the performance indices on the shear rates at the beginning and end of the lotion application (γ_1 and γ_2), which are parameters that describe the way the customer uses the product. Thus, in a way, usage functions are integrated in these property functions.

Step 3. Develop a process function

A batch process is considered (Figure 3) comprehending the following operations [10]:

1. In tank T-1: (i) charge part A; (ii) dissolve solids, heat and mix;
2. In tank T-2: (i) charge part B; (ii) heat and mix; (iii) add part A; (iv) cool and mix (pre-emulsion formation); (v) add part C and mix;
3. Homogenization in colloid mill CM-1 (continuous operation);
4. Filling and packaging.

A model for the process is developed, including mass and energy balances, droplet breakage relationships predicting the oil droplets' size [11, 12], kinetic models predicting the heating, cooling and mixing times and also some considerations about process scheduling. The overall process model has the following structure: inputs – product design variables (w_T, w_G, ϕ), annual production (AP), production per batch (PB), equipment dimensions, operating temperatures and mixing rates; outputs – operating times, consumed energies, number of batches per year (NB), annual operating time (AOT), effective batch time (EBT) and maximum droplet diameter (D_{max}). A relationship between the maximum droplet diameter, which is a structural attribute of the product, and process design and operation variables is incorporated in the model. This relationship is, in the language of the chemical product pyramid, a process function.

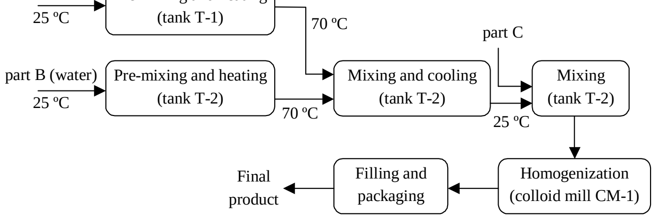

FIGURE 3
LOTION MANUFACTURING PROCESS.

Step 4. Integrate product and process design

The integrated product/process design problem is formulated as follows: given the annual production required, find the product composition (w_T, w_G, ϕ), production per batch (PB), equipment dimensions and mixing rates that minimise an overall annual cost, equal to the sum of investment, production and quality loss costs. Quality costs are considered proportional to the total loss ($L_1 + L_2$) provoked by deviations in the performance indices μ_1 and μ_2, and estimated as 30% of the production costs when ($L_1 + L_2$) = 25%.

The restrictions in the optimisation formulation include the property, usage and process functions discussed above and the condition related to the acceptance of the lotion in terms of smoothness ($D_{max} \leq 10$ μm).

The optimisation problem is implemented and solved using GAMS/CONOPT3. For an annual production AP = 2000 ton/yr, the results of Table III are obtained. The non-null quality cost (2.0 thousand Euro/yr) means that the product performance indices (μ_1 and μ_2) are somehow sacrificed in order to obtain an optimal product/process performance that takes into account both product quality and process costs.

The benefits associated with this integrated solution may be assessed by comparing it with a decoupled solution. When the product design problem is solved separately, one obtains a product composition ($w_T = 0.8515$, $w_G = 12.0$ and $\phi = 16.67$) that perfectly matches the target specifications μ_1* and μ_2* and thus the quality cost is null. Then, designing the process to manufacture this product, one obtains a total cost (investment plus production) of 335.4 thousand Euro/yr, which is 2.4 thousand Euro/yr greater than the total cost for the integrated solution. This difference (which increases when the limit of 10 μm in D_{max} is lowered) clearly illustrates that a decoupled sequential approach to product and process design may lead to suboptimal solutions, reinforcing the importance of adopting an integrated perspective.

TABLE III
OPTIMAL PRODUCT/PROCESS DESIGN SOLUTION.

Product design variables	
w_T = 0.8454 % (w/w)	w_G = 12.00 % (w/w)
ϕ = 18.13 % (v/v)	
Product performance indices	
μ_1 = 375.0 Pa.s	μ_2 = 0.0256 Pa.s
D_{max} = 10 μm	
Process design and operation variables	
PB = 3429 kg/batch	EBT = 280 min/batch
NB = 583 batch/year	AOT = 2724 h/year
Tank T-2 (*)	*Colloid mill CM-1* (*)
D = 1.63 m	D = 0.121 m
P = 12.5 kW	P = 1.91 kW
N = 117 rpm	N = 3600 rpm
Costs (thousand Euro/yr)	
Investment	86.8
Production	244.2
Quality	2.0
Total	333.0

(*) D, P and N represent diameter, mixing power and mixing rate.

CONCLUSIONS

Chemical product engineering as a systematic discipline is still evolving and there is not yet a consensus about an appropriate framework that would be essential namely to establish efficient teaching strategies and practices. The operational facet of the discipline is chemical product design that should be in part taught and learnt based on case studies, due to its practical nature as well as the wide variety of products that chemical engineers have to deal with. In this regard, the web resource of examples here presented is a valuable contribution. Furthermore, the website is designed according to a generic product design template [1] and also incorporates some concepts that have been recently proposed specifically for chemical products [2]. Therefore, it is also a valuable contribution to a more methodical teaching of the discipline.

Regarding the particular case study that has been presented in more detail, through it general concepts of chemical product design (such as property, usage and process functions) become clearer, and the relevance of specific questions, namely the importance of microstructure in formulated consumer products and the interaction between product and process design decisions, is highlighted. Furthermore, several scientific topics (rheology, emulsification) and engineering tools (mechanistic and statistical modelling, optimisation tools) are managed, integrating knowledge from chemical engineering core courses. In this manner, it has also been illustrated how learning chemical product design through case studies may be efficient to consolidate previously addressed scientific and technical topics.

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