# Optimization of an Alkylpolyglucoside-Based Dishwashing Detergent Formulation

**I. Bozetine · T. Ahmed Zaïd · C. E. Chitour · J. P. Canselier**

**Abstract** The aim of this work was to formulate and optimize the washing performance of an alkylpolyglucoside-based dishwashing detergent. The liquid detergent was formulated with five ingredients of commercial origin: anionic (linear sodium alkylbenzenesulfonate and sodium laurylethersulfate), nonionic ($C_{12}$–$C_{14}$ alkylpolyglucoside) and zwitterionic (a fatty acid amide derivative with a betaine structure) surfactants, and NaCl for viscosity control. In addition to the plate test, other properties were investigated including "cloud point", viscosity, and emulsion stability. Statistical analysis software was used to generate a central composite experimental design. Then, a second order design and analysis of experiments approach, known as the Response Surface Methodology, was set up to investigate the effects of the five components of the formulation on the studied properties in the region covering plausible component ranges. The method proved to be efficient for locating the domains of concentrations where the desired properties were met.

**Keywords** Alkylpolyglucoside · Dishwashing detergent · Formulation · Response surface methodology · Washing performance

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## Introduction

Liquid detergents play very important roles in our daily lives for personal care, household surface care, and fabric care. For many reasons, mainly because they dissolve more rapidly than powdered detergents and are easier to dose, liquid detergents have gained an increasing popularity [1].

All other factors, surface to be cleaned, soil, water hardness, and temperature—being equal, cleaning performance is a function of the concentrations and types of the active ingredients that are delivered into the cleaning bath. Among these active ingredients, the nonionic surfactants alkylpolyglucosides (APGs) have been known for a long time [2, 3]. As a result of their interesting properties and of the development of new and efficient technologies, they have been incorporated into detergents, namely dishwashing liquids, since the 1990s [4–9]. APGs are currently manufactured by several large detergent companies [10, 11]: they are derived from renewable raw materials, namely glucose and fatty alcohols (coming from vegetable oils). Their physical-chemical properties in pure aqueous solution [10, 12–14] or mixed with other surfactants [15, 16], then often showing synergistic effects [6, 17], have been extensively investigated. In addition to their mildness to the skin, APGs are readily and rapidly biodegradable when discharged into the aerobic aquatic environment [18, 19] and even possess detoxifying properties [20]. They are among the most resistant non-ionic species to alkaline hydrolysis [5, 20].

In the present study, the goal was to formulate a hand dishwashing detergent with four major ingredients of commercial origin.

Statistical analysis software was used to generate a central composite experimental design and to investigate the effects of the five components on the following selected

properties: washing performance according to ISO 4198 standardized procedure [21], viscosity, cloud point and emulsion stability. Besides good washing performances, a viscosity of at least 300 cSt and a cloud point lower than 5 °C were desired. Only quantitative aspects of the formulation, on a physical chemical basis, were considered, although psychological and economical aspects may also be important regarding the detergent market.

## Experimental

### Materials

*Ingredients*: Except for NaCl (analytical grade), all the ingredients: anionic, nonionic and zwitterionic surfactants of commercial origin (Henkel-Cognis GmbH), were used without further treatment.

The trade names, chemical compositions, physical states and approximate costs of the ingredients are given in Table 1.

*Soil*: Mixture of vegetable (palm and sunflower) oils (product of *Cevital*), chosen according to local culinary habits.

*Surface*: 20 cm diameter dinner plates.

### Methods

Experimental procedures are very close to those described in [22] with other components. The formulas were prepared in 100 g quantities. Ingredients were mixed in the increasing order given in Table 1 at room temperature and homogenized after addition of distilled water up to 100 g. The pH-value was adjusted to 6.5–7.5 by addition of citric acid.

- The dishwashing test was carried out according to the ISO 4198 guide for comparative testing of performances [21]. The result of the test was taken as the number of plates, $N_p$, which could be cleaned until the "foam end point" was reached, that is when half the surface of the water was covered with a thin layer of foam. Usually, the numbers of plates washed when liquor not covered with foam becomes visible and when almost complete destruction of foam occurs are $N_p - 1$ and $N_p + 1$, respectively. This so-called "foam end point" is considered as the point least subject to errors in judgment by operators [1, 23]. A few parameters and conditions need to be specified:

*Preparation of soiled plates*: 150 µL of vegetable oil were transferred to a dry, clean ceramic plate using an automatic pipette. The oil was then spread uniformly on the plate.

*Preparation of dishwashing solution*: 2.4 mL of liquid detergent were first transferred to a 35 cm diameter wash pan of plastic material. Three liters of water (280 ppm hardness) were then poured into the pan using a 1 L glass flask from a height of 20 cm above the center of the pan in order to produce the initial foam. A detergent concentration of 0.8 mL/L was thus used. The dishwashing solution temperature was approximately 25 °C.

*Washing procedure*: A 7 cm × 10 cm dish mop was used for this operation. The soiled dishes were washed one at a time, both front and back, using a rotating motion with the dish mop while keeping the dish half submerged in a given angular position (ca. 45°) with respect to the bottom of the wash pan. This wash process took around 30 s, including the rinsing step and the visual inspection of the plate. The plates that had been washed and rinsed during the test were placed in racks and allowed to dry.

- Kinematic viscosity ($v$) was measured at 25° using Ubbelohde viscometers.
- The "cloud point", $t_{cloud}$, was determined by cooling the samples in 20-mL glass test tubes in an ice bath and the appearance of turbidity was taken as the cloud point. The reproducibility was ±1 °C. The "clear point", $t_{clear}$, was observed when the sample became transparent while coming back to room temperature. In

**Table 1** Ingredients of formulated liquid detergents and their physical states and approximate costs

| | Compound (commercial name) | Chemical structure, class (concentration in wt.%, physical state) | Current cost (€/kg approximate) |
|---|---|---|---|
| 1 | Maranil® Paste A55 | Linear sodium dodecylbenzenesulfonate, anionic surfactant (54–56% pasty) | 0.55 |
| 2 | Texapon® NSO | Sodium laurylethersulfate, anionic surfactant (26.5–27.5% liquid) | 0.40 |
| 3 | Glucopon® 600CS UP | Alkylpolyglucoside, nonionic surfactant (50–53% thick liquid) | 1.65 |
| 4 | Dehyton® K | Fatty acid amide derivative with betaine structure, zwitterionic (29–32% pasty) | 0.70 |
| 5 | Sodium chloride | NaCl (crystalline solid) | 0.28 |

Europe and North America, recommended values of "cloud point" and "clear point" are lower than 5 °C and lower than 10 °C, respectively [1].

- The emulsifying power of the samples towards grease (emulsion stability) was illustrated by the following test. 20 g of a solution of known composition were introduced into a 30-mL vial (2.5 cm × 9.5 cm) and 0.2 g of vegetable oil was added to the vial. The vial was stoppered and submitted to an oscillatory motion (180° back and forth) twenty-five times at an approximate rate of one rotation per second. The vial was then permitted to stand for 5 min. Nephelometric readings (intensity of the light scattered at 90°) were taken using a WTW Turb 555 turbidimeter after 1, 3 and 5 min. and expressed in NTU. Linear regressions gave the predicted readings for 3 min, which were taken as the turbidity values, $\tau$, higher for more stable emulsions [24].

**Table 2** Concentrations of ingredients

| Compound: commercial name, abbreviation | Concentrations: reduced and actual (wt.%) | | | | |
|---|---|---|---|---|---|
| | −2 | −1 | 0 | +1 | +2 |
| Maranil® Paste A55, M | 4 | 7 | 10 | 13 | 16 |
| Texapon® NSO, T | 4 | 6 | 8 | 10 | 12 |
| Glucopon® 600CS UP, G | 0 | 2 | 4 | 6 | 8 |
| Dehyton® K, D | 0 | 1 | 2 | 3 | 4 |
| NaCl, NaCl | 0 | 0.3 | 0.6 | 0.9 | 1.2 |

## Statistical Methods

The second-order experimental design, a central composite design with four center points, is shown in Table 3. All

**Table 3** Composition and measured properties of samples for the central composite design

| Run N° | Ingredients and reduced concentrations | | | | | Response variables | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | M | T | G | D | NaCl | $N_p$ | $v$ (cSt) | $\tau$ | $t_{cloud}$ (°C) | $t_{clear}$ (°C) |
| 1 | −1 | −1 | −1 | −1 | 1 | 15 | 11 | 23 | 2 | 8 |
| 2 | −1 | −1 | −1 | 1 | −1 | 18 | 8 | 13 | 0 | 6 |
| 3 | −1 | −1 | 1 | −1 | −1 | 25 | 15 | 16 | −1 | 7 |
| 4 | −1 | −1 | 1 | 1 | 1 | 25 | 581 | 10 | −2 | 9 |
| 5 | −1 | 1 | −1 | −1 | −1 | 19 | 9 | 18 | 0 | 5 |
| 6 | −1 | 1 | −1 | 1 | 1 | 36 | 24 | 11 | −1 | 5 |
| 7 | −1 | 1 | 1 | −1 | 1 | 35 | 112 | 13 | −3 | 8 |
| 8 | −1 | 1 | 1 | 1 | −1 | 42 | 35 | 18 | −4 | 7 |
| 9 | 1 | −1 | −1 | −1 | −1 | 24 | 10 | 16 | 2 | 15 |
| 10 | 1 | −1 | −1 | 1 | 1 | 29 | 46 | 17 | −3 | 6 |
| 11 | 1 | −1 | 1 | −1 | 1 | 28 | 169 | 17 | 0 | 9 |
| 12 | 1 | −1 | 1 | 1 | −1 | 33 | 45 | 15 | −1 | 7 |
| 13 | 1 | 1 | −1 | −1 | 1 | 32 | 18 | 11 | 0 | 16 |
| 14 | 1 | 1 | −1 | 1 | −1 | 29 | 12 | 16 | −2 | 6 |
| 15 | 1 | 1 | 1 | −1 | −1 | 43 | 20 | 17 | −3 | 7 |
| 16 | 1 | 1 | 1 | 1 | 1 | 47 | 442 | 13 | −2 | 7 |
| 17 | −2 | 0 | 0 | 0 | 0 | 25 | 19 | 17 | −1 | 8 |
| 18 | 2 | 0 | 0 | 0 | 0 | 28 | 32 | 23 | −1 | 7 |
| 19 | 0 | −2 | 0 | 0 | 0 | 22 | 23 | 12 | −1 | 7 |
| 20 | 0 | 2 | 0 | 0 | 0 | 33 | 22 | 15 | −2 | 6 |
| 21 | 0 | 0 | −2 | 0 | 0 | 39 | 10 | 17 | −2 | 14 |
| 22 | 0 | 0 | 2 | 0 | 0 | 29 | 419 | 13 | 0 | 7 |
| 23 | 0 | 0 | 0 | −2 | 0 | 31 | 12 | 15 | 0 | 7 |
| 24 | 0 | 0 | 0 | 2 | 0 | 35 | 135 | 8 | −1 | 7 |
| 25 | 0 | 0 | 0 | 0 | −2 | 22 | 10 | 18 | −4 | 6 |
| 26 | 0 | 0 | 0 | 0 | 2 | 23 | 63 | 14 | −3 | 6 |
| 27 (C) | 0 | 0 | 0 | 0 | 0 | 31 | 24 | 13 | −2 | 6 |
| 28 (C) | 0 | 0 | 0 | 0 | 0 | 30 | 26 | 14 | −3 | 8 |
| 29 (C) | 0 | 0 | 0 | 0 | 0 | 27 | 23 | 13 | −2 | 6 |
| 30 (C) | 0 | 0 | 0 | 0 | 0 | 26 | 24 | 13 | 0 | 6 |

analyses were performed with the help of Statistica® (Design of Experiment module) [25], which provides a set of options to allow the user to optimize multiple response variables interactively, given the current model. For convenience, analyses were performed using coded independent variables (−2, −1, 0, +1, +2) rather than actual values. In model quadratic equations, only some of the terms are statistically significant. The Pareto diagram sorts the effect (absolute values) of the parameters (independent variables) on the responses (dependent variables). Unless otherwise stated, the threshold value was fixed at 5% ($p < 0.05$).

## Results and Discussion

The actual and corresponding reduced concentrations of the ingredients are reported in Table 2. Along with the reduced concentration values of the five ingredients, the response variables (number of plates washed, viscosity, turbidity, cloud point and clear point) are indicated in Table 3.

*Plate test*: For a 5%, threshold on the Pareto chart, only Texapon, with its linear term, shows a significant effect on $N_p$. As can be seen for $p = 0.15$, Texapon, Glucopon and Maranil exhibit a statistically significant effect on the number, $N_p$, of cleaned plates (Fig. 1). The model for this response is:

$$N_p = 29.37 + 4.50T + 2.33G + 2.33M \quad (1)$$

with a rather low correlation coefficient $R^2$ of 0.47. In fact, a lot of factors, associated with the details of the experimental procedure and which cannot be accounted for, affect detergent performance. For instance, the exact quantity of soil deposited cannot be known with high precision as some soil is always left on whichever device is used to spread it onto the plate. This in turn will have an effect on the foaming power and hence the plate number.

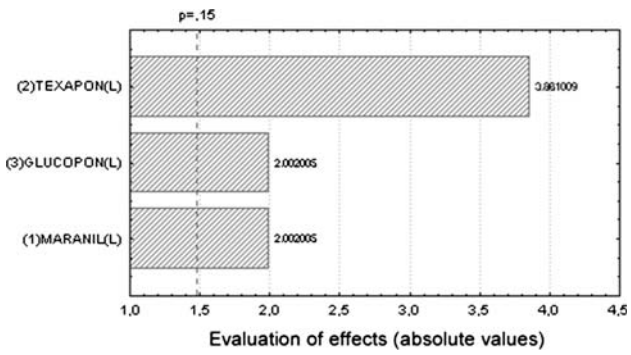


**Fig. 1** Pareto chart of effects for the plate test (*L* stands for linear)

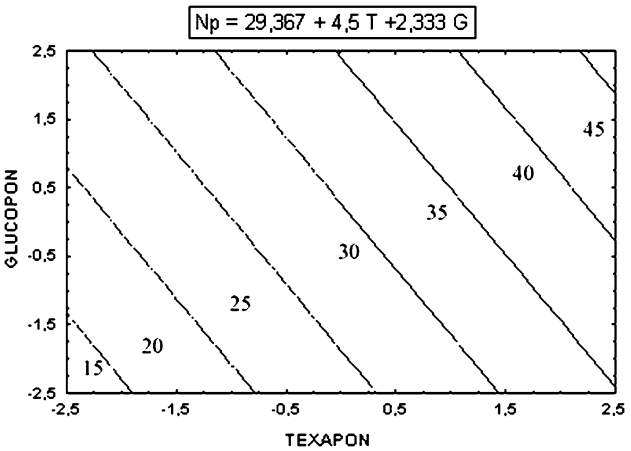


**Fig. 2** Contour plots for predicted number of plates (M = D = NaCl = 0)

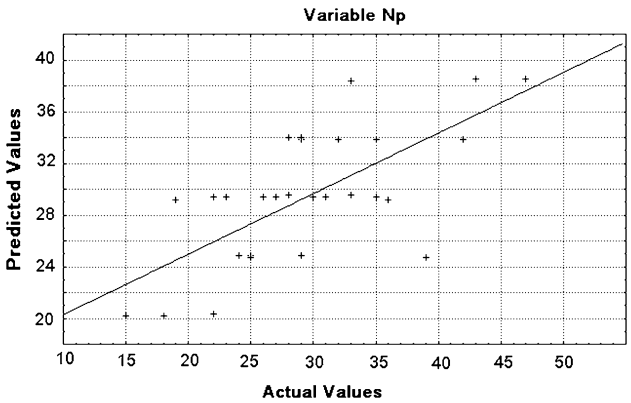


**Fig. 3** Predicted versus actual values for number of plates ($N_p$)

The contour plots for $N_p$ (projection of the response surface) predicted from the model are given in Fig. 2. From this plot, one can easily see that the best washing performances are obtained with increasing surfactant concentrations.

The precision of the $N_p$ model can be best visualized by a plot of actual versus predicted values (Fig. 3).

Residuals are differences between the observed values and the corresponding ones predicted by the model and thus represent the variance that is not explained by the model. The better the fit of the model, the smaller the values of residuals. Figure 4 shows the residuals plot.

Analysis of variance for the $N_p$ model is given in Table 4.

Obviously this result is specific to a particular application. Since there is wide variation in soils and consumer dishwashing habits, for a given detergent formula, the use of other types of soil (e.g. those described in the ASTM D4009 standardized procedure) [23] and washing procedure would lead to different values of $N_p$. It is not likely that a general equation could be obtained to represent the

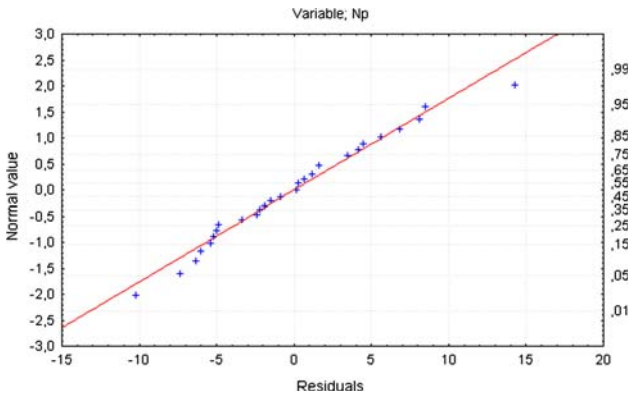


**Fig. 4** Residuals plot for the number of plates ($N_p$)

**Table 4** Anova table for the number of plates ($N_p$)

| | Sum of squares | Degrees of freedom | *F*-value | *p*-value |
|---|---|---|---|---|
| (1) Maranil® (L) | 130.667 | 1 | 23.05882 | 0.017189 |
| (2) Texapon® (L) | 486.000 | 1 | 85.76471 | 0.002664 |
| (3) Glucopon® (L) | 130.667 | 1 | 23.05882 | 0.017189 |
| Lack of fit | 830.633 | 23 | 6.37315 | 0.075848 |
| Pure error | 17.000 | 3 | | |
| Total sum of squares | 1,594.967 | 29 | | |

performances of a family of detergents containing various ratios of the same ingredients.

*Viscosity* The viscosity of a liquid detergent is very important for its dispersability on dilution [26]. The viscosity of a light duty liquid detergent is typically in the range of 100–500 cSt.

At $p = 0.05$ level, three ingredients were found to show significant effects on viscosity (Fig. 5).

Excluding the other parameters, the viscosity can be described by the following equation:

$$v = 42.34_5 + 87.44\mathrm{G} + 46.94\mathrm{G}^2 + 56.32\mathrm{NaCl} + 46.58\mathrm{G.D} + 70.55\mathrm{G.NaCl} + 46.07\mathrm{D.NaCl} \quad (2)$$

with a correlation coefficient $R^2$ of 0.92.

The contour plots of predicted viscosity from the above model are given in Fig. 6. Viscosity values reach 300 cSt and more in the upper right corner of the plot (high concentrations of Glucopon and NaCl).

*Emulsion Stability*. Turbidity measurements were performed in order to measure product ability to emulsify grease (see "Experimental").

At $p = 0.05$ level, the Pareto and contour charts for turbidity are given in Figs. 7 and 8, respectively. The model equation for turbidity reads:

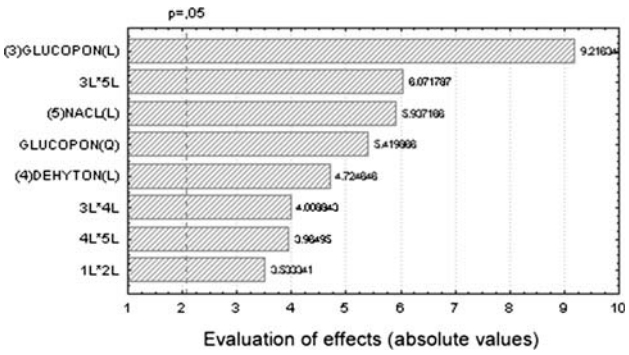


**Fig. 5** Pareto chart of effects on viscosity (*L* stands for linear and *Q* for quadratic)

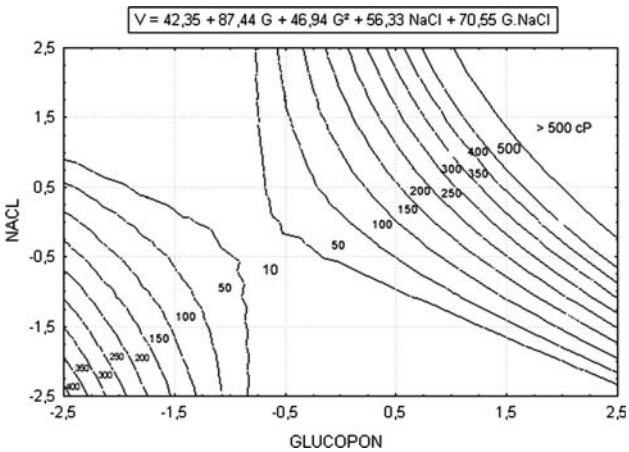


**Fig. 6** Contour plots of predicted viscosity (M = D = T = 0)

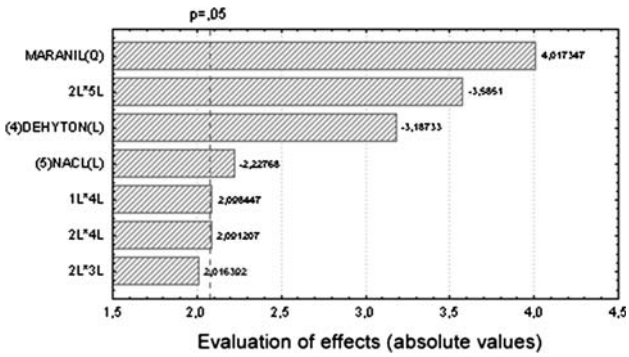


**Fig. 7** Pareto chart of effects on turbidity (*L* stands for linear and *Q* for quadratic)

$$\tau = 13.82 + 1.55\mathrm{M}^2 - 1.35\mathrm{D} + 1.09\mathrm{M.D} + 1.08\mathrm{D.T} - 0.94\mathrm{NaCl} + 1.04\mathrm{T.G} - 1.86\mathrm{T.NaCl} \quad (3)$$

with a coefficient of determination $R^2$ of 0.72.

Turbidity does not vary very much throughout the investigated concentration range. The higher (desirable)

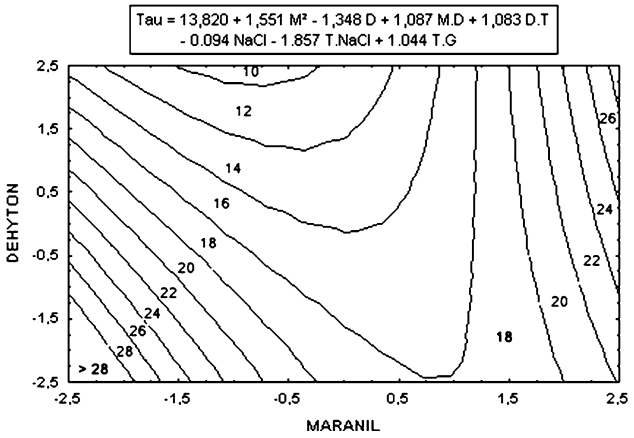


**Fig. 8** Contour plots of predicted turbidity (T = G = NaCl = 0)

values obtained at the lower left corner of the plot, corresponding to low concentrations of Maranil and Dehyton (out of the range studied) are not reliable. Therefore, the emulsifying power will not be critical for the formulation.

*Cloud point*: The cloud point is the temperature at which the product begins to turn cloudy upon cooling. At $p = 0.1$ level, only two ingredients were found to exhibit significant effects on the cloud point. The contour chart for the cloud point is given in Fig. 9. The model for the cloud point can be described by the following equation:

$$t_{\text{cloud}} = -1.33 - 0.58\text{T} - 0.58\text{D} \quad (4)$$

with $R^2 = 0.73$.

In fact, in the investigated concentration range, the products should exhibit cloud points lower than 0 °C. On the other hand, except for samples 9, 13 and 21, the clear points do not exceed 10 °C.

## Conclusion

Finally, we may conclude that:

- The liquid detergent we have formulated possesses acceptable properties nearly throughout the investigated composition range,
- Practically, all the properties improve for higher surfactant concentrations (of course, at the expense of cost).
- Emulsion stability (expressed as turbidity) and cloud point are not very critical. When choosing the optimum for the product formulation, the two main properties to be considered are the cleaning properties and the viscosity.

Obviously, the active matter concentration of the finished detergent is probably the most important parameter as it has a direct influence on both physical and performance characteristics. The active matter concentration also, of course, determines the cost of the finished formulations. In the present case, contour plots and desirability profiles help to achieve the best solution.

**Table 5** APG-based dishwashing detergent: limit and compromise formulas

| Ingredient | Concentrations (wt.%) | | | |
|---|---|---|---|---|
| | In limit formula | | In compromise formula | |
| | Reduced | Actual | Reduced | Actual |
| Maranil® Paste A55 | 1 | 13 | 0 | 10 |
| Texapon® NSO | 1 | 10 | 0.5 | 9 |
| Glucopon® 600CS UP | 1 | 6 | 0.5 | 5 |
| Dehyton® K | 1 | 3 | 0 | 2 |
| NaCl | 1 | 0.9 | 1 | 0.9 |
| Property | | | | |
| $N_p$ (plate number) | 38.5 | | 32.8 | |
| $\nu$ (cSt) | 396 | | 190 | |
| $\tau$ (NTU) | 14.44 | | 12.21 | |
| $t$ (°C) | −2.5 | | −1.6 | |

The addition of NaCl increases the viscosity of the mixture, showing the same trend as the addition of surfactants (Eq. 2). So, from Eqs. 1 to 4, it is interesting to calculate the properties (responses) of the detergent formulated according to the experiment n° 16, where all the ingredients are at their maximum concentrations, i.e. +1 in reduced value (Table 2). The obtained values, reported in Table 5, largely exceed the initial fixed goals ($\nu \geq 300$ cSt, $Np \geq 30$ and $t_{\text{cloud}} \leq 5$ °C), but correspond to a high ratio of active matter ($\sim 14\%$) and to the highest cost (within the limits defined above): 0.234 €/kg.

In order to reduce ingredient consumption, it will be advantageous to lower the surfactant amounts while maintaining NaCl (cheapest component) at its highest level so that viscosity remains high. Keeping the fixed goals in mind with regard to cleaning power, a second, "compromise formula", together with the properties herewith associated, is presented in Table 5. With T = G = 0.5, M = D = 0, therefore ca. 11% active matter, except for the kinematic viscosity, the properties appear satisfactory at a lower cost of 0.19 €/kg. To reach the desired viscosity, it would be sufficient to increase the amount of NaCl up to 1.26% (reduced value: 2.21, making the hypothesis that our model remains valid). Emulsion stability would be a little less ($\tau = 10$ NTU) and the cost would then be 0.194 €/kg, slightly higher than the previous one.

The method proved to be efficient for sketching out the domains of concentrations where the desired properties are met. Unfortunately, the models should probably not be applied to similar products from different suppliers. It is well known that the chemical composition of commercial

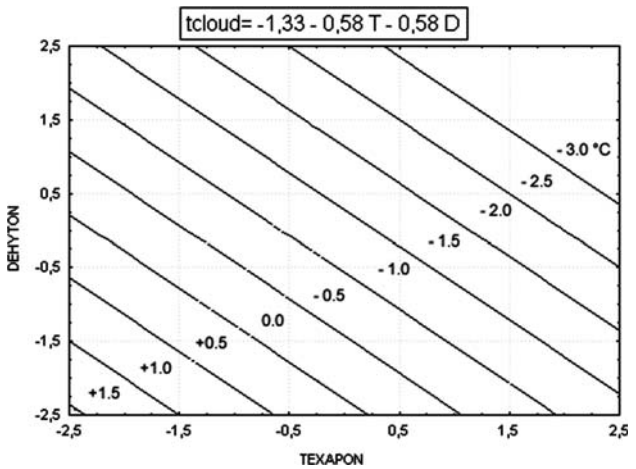


**Fig. 9** Contour plots of predicted cloud point (M = G = NaCl = 0)

surfactants (which are always mixtures) varies with the details of the manufacturing process and even from batch to batch, although the global specifications are met. Finally, let us note that this choice does not take ingredient cost as an optimization criterion and should be modified if a quality-to-cost optimum formulation was searched for.

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**Jean Paul Canselier, D. Sc.,** just retired, has been a Lecturer at the School of Chemistry and Process Engineering of Toulouse (ENSIACET). He has more than 25 years experience in the domain of surfactants and dispersed systems (mixed micelles, microemulsions, surfactants in chemical engineering operations, formulation, etc.). He has authored or co-authored around seventy papers and book chapters, mainly in this research area.