Bath-substrate-flow method for evaluating the detersive and dispersant performance of hard-surface detergents

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Bath−Substrate−Flow Method for Evaluating the Detersive and Dispersant Performance of Hard-Surface Detergents

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A method has been formulated and tested in a laboratory device to evaluate the detersive and dispersant performance, as well as the washing dynamics, of surfactants and builders and the formulation of the detergent. The method, called bath−substrate flow (BSF), is based on the separation of the washing bath from the substrate to be washed after contact of the two by a cyclic-flow process. The method serves to analyze mechanical washing systems, such as dishwashers, tunnel washes, floor-washing machines, and industrial-scale cleaning systems. The circumscribed central compound experimental design was applied, as was the response surface method, to correlate the detergency values found.

1. Introduction

Detergency is difficult to evaluate because it depends on a multitude of variables that in most cases are elusive to monitor and measure.[1] The tests can be classified into three groups: laboratory tests, practical evaluation, and consumer reaction. If a commercial product is to be evaluated, these types of tests should be made successively.

Laboratory tests should attempt to reproduce real conditions. Furthermore, they should detect the truly significant variables and monitor and measure them accurately. If these conditions are met, the laboratory tests are useful for research into the performance of an established detergent formula or a new one under formulation.

The basic steps to follow are (a) choice of the substrate and the soils; (b) soiling of the substrate in the most reproducible way possible; (c) washing of the substrate by contact with a wash bath while controlling the experimental conditions; (d) removal of the substrate from the bath (or vice versa) and analysis of how much soiling was removed or how much was transferred to the wash bath.

Currently, for textile detergents, a large array of methods are available to evaluate the detersive capacity of a product. These methods are widely used, as is advisable in several standardized detergency-testing methods, such as those offered by the American Society for Testing and Materials (ASTM) or the International Standardization Organization (ISO), with its Spanish equivalents (UNE).

On the other hand, in the case of tests for dishwashing detergents for machines, no methods or laboratory devices are available, and only practical evaluations are performed.

In a generic way for hard surfaces, elemental methods are normally used: simply soiling a small plaque of the study material and submerging it slowly into a bath, leaving half unsubmerged. After a specified time, it is removed and the detersive capacity is evaluated by comparing the differences between the halves. Immersion should be performed slowly by the use of a small electric motor to achieve adequate reproducibility.[2]

Another method is that of the rotating disk, consisting of spinning a soiled Teflon disk in a tank containing a cleansing solution. By an appropriate analytical method applied to the bath, the detersive effect is determined. Also, microphotographic studies can be made of the disk surface.[3−7]

In the case of cleaners that are applied by a spray gun, a device consisting of a rotating sprayer that projects the detergent solution onto small soiled plaques has been designed. The soiled liquid that falls is collected and analyzed. One noteworthy result was to find the optimal detergency at surfactant concentrations up to more than 100-fold higher than the critical micellar concentration (cmc), with this contradicting previously published data, which placed the optimal cleansing values at around the cmc values.[8]

These methods present the problem that the surface treated is of reduced dimensions and the evaluation of the detergency can be difficult, and one serious disadvantage is that it is not possible to determine the kinetics parameters of the exchange soiling/bath in the detersive process. In addition, as demonstrated below, these methods do not accurately simulate the functioning of the washing by machines used on hard surfaces (dishwashers, tunnel washers, floor washers, etc.).

Table 1 presents some of the standardized tests that use the detergency-evaluation methods cited.

After the washing, it is indispensable to evaluate the detersive result, determining the amount of soiling agent removed from the surface or washed off into the bath. The methods used can be classified as optical, gravimetric, radiotracing analysis, etc.

1.1. Description of the Bath−Substrate-Flow (BSF) Method. The method proposed,[9] called the BSF method, as demonstrated below, is useful for laboratory evaluations of the detersive capacity of surfactants, detergency builders, and detergent compositions for hard surfaces. It consists of the following:

(a) The substrate or surface to be washed should present a large specific surface. For this, the substrate can be divided into small sections, preferably of well-

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Table 1. Examples of Standardized Tests for Hard-Surface Detergency

organization	method code	method title
ASTM	D 3556	standard test method for deposition on glassware during mechanical dishwashing
ASTM	D 3565	standard test method for tableware pattern removal by mechanical dishwasher detergents
ASTM	D 5343	standard guide for evaluating the cleaning performance of ceramic-tile cleaners
ASTM	D 6215	standard guide for removal of oily soils from metal surfaces
ASTM	D 4488	standard guide for testing the cleaning performance of products intended for use on resilient flooring and washable walls
ISO	4198	surface-active agents—detergents for hand dishwashing—guide for comparative testing of their performance
ISO	7535	surface-active agents—detergents for domestic machine dishwashing—guide for comparative testing of their performance
UNE	55829:1986 (equivalent to ISO 4198)	surface-active agents—detergents for hand dishwashing—guide for comparative testing of their performance
UNE	55828:1986 (equivalent to ISO 7535:1984)	surface-active agents—detergents for machine dishwashing—guide for comparative testing of their performance

defined geometric shape, particularly in the form of spheres, cubes, cylinders, prisms, tubes, or rings, because these enable easy determination of the total area exposed to the washing. The use of a large surface enables the use of large quantities of soil in a compact system of small dimensions, thereby improving the precision of the analytical method used to evaluate the detergency.

(b) The substrate can be of any nature, provided that it is sufficiently hard, such as glass, ceramics, porcelain, metal, or plastic.

(c) There are no restrictions on the type of liquid or solid soil, although the results prove better with food types, such as lipids, carbohydrates, proteins, and colorants, with the most usual of the latter being coffee and tea. In addition, other types of soil are used, such as lipstick or soil common on floors, especially hydrocarbon or particles of clay, limestone, oxides, etc.

(d) The cleaning solution and the substrate to be washed are kept separate, allowing contact by flow by not by immersion.

(e) According to this flow method, the washing consists of the following: on an impulsion system, washing is cyclic and continuous, during which the cleaning solution makes contact with the substrate and removes part of the soil, and the resulting soiled water returns to the container with the rest of bath, where it is homogenized to begin a new cycle. As shown in the scheme of Figure 1, 1 represents the bath, 9 is any mixing device that keeps the bath homogeneous, 2 is any impulsion system, and 3 is the column or tank that contains the substrate, divided according to the specifications in part a. Both the bath and the packed column must have a temperature-control system, 4 and 5, it being advisable also for the heat losses in the tubes 7 and 8 to be minimized. In addition, there should be a flow control on the system, 6.

(f) Detergency can be evaluated either from the soil remaining on the substrate or from the soil washed into the bath, although it is preferable to analyze the bath. Any analytical method normally employed in other evaluations of detersive capacity (cited above) can be used, except those by eye or by surface reflectance. On the other hand, many other less usual methods may be preferable, being feasible because of the greater quantity of soiling agent applicable in the test, given the substantial increase in the substrate surface area. For example, grease soil can be identified by evaluating the fatty acids with alkali or by saponification of esters. Starches can be determined by specific analyses and proteins by total-N determination, among other techniques. In general, detergency can be appraised either by sampling the bath, 10, or by using a measurement system of a physicochemical variable related to detergency, 11. The latter option enables easy dynamic monitoring of the detersive process.

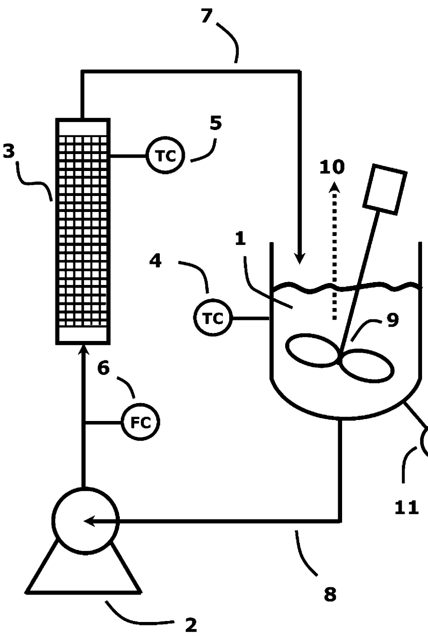

Figure 1. Scheme of the BSF method.

(g) The soil can easily be applied uniformly by vigorously mixing it with pieces of the substrate. For example, the substrate and soil can be placed in a closed container and stirred in all directions with a prefixed strategy. After this operation and, if desired, after an aging process, the column or the tank with the substrate is filled, to proceed to the washing as described in parts d and e.

It should be emphasized that this method to evaluate the detersive effectiveness, based on the bath−substrate separation, is completely novel with respect to any other system previously proposed, simulating moreover a multitude of mechanical cleaning systems in which this separation also occurs. As opposed to textile washing, in which the fabrics are completely submerged in the cleaning solution, maintaining complete substrate−bath contact by immersion, the present device offers progressive contact by flow. In addition, the surface area soiled is very large.

Figure 2 shows a simplified scheme of a dishwasher, indicating that these machines act as a system with a bath−substrate separation. These appliances, both do-

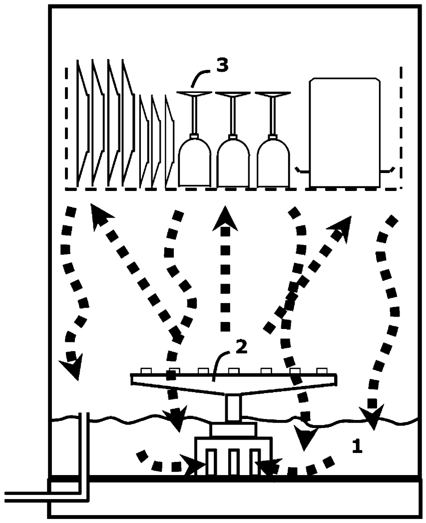

Figure 2. Scheme of a dishwasher.

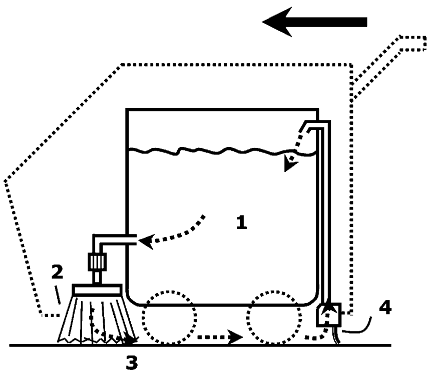

Figure 3. Scheme of a floor washer.

mestic and industrial have, in general terms, a lower tank, 1, which contains the bath and a system of rotating washer blades, 2, which spray the detergent solution onto the dishes placed in the upper part in a basket, 3. This cleaning system therefore consists of creating an ascending and descending flow simultaneously between the bath and the substrate being washed, the two being physically separated. In addition, as in the case of the machines made for restaurants, with practically the same bath, successive loads of dishes are washed, and therefore the total soiled surface area treated is very large. The washing is complemented by a final water rinse and an auxiliary rinse or a rinse for shine. The kinetics of the detersive process has, apart from the influence of the detergent and temperature, a temporal component, the time needed to apply the cleaning solution to the substrate and the subsequent return of the soiled solution to the bath. The laboratory methods based on the rotation of disks submerged in the bath or those based on immersing a plaque into a stirred container are not capable of evaluating this dynamic behavior presented in reality by dishwashers and other washing machines discussed below.

Figure 3 presents another example of substrate−bath separation and contact by flow for washing large surfaces, with this scheme depicting a floor washer. As opposed to the dishwasher in which both the surface and the machine maintain their positions relatively constant, here the machine must move over a stationary substrate. Basically, the washers consist of a vehicle with wheels, with a tank containing the bath, 1, rotating brushes, 2, which become soaked with the cleaning solution to scrub the floor, 3, and in back the vacuum system, 4, for collecting the soiled liquid from the floor and returning it to the bath. In this system also, the bath and substrate are separated and the flow occurs by injection and subsequent vacuuming.

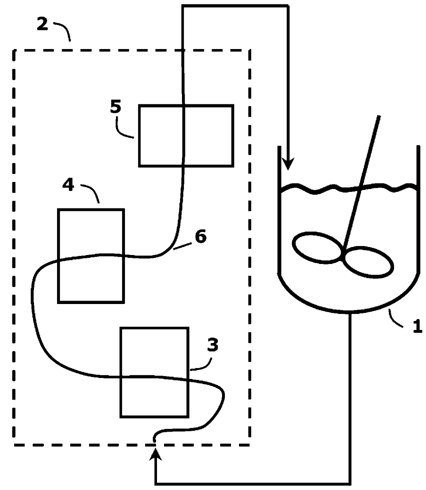

Figure 4. Scheme of a CIP cleaning process.

Another noteworthy example (Figure 4) is the flow process of cleaning in place (CIP), in which an industrial circuit, 2, is cleaned. The circuit can be any installation, although preferably of a agrofood type: automatic milking systems for cows and goats, milk-treatment machinery, beer brewery, carbonated-drink factory, etc. In all of these cases, a tank contains the cleaning solution 1, which passes repeatedly through the entire installation, 2, washing both the pipes, 6, and the equipment, 3−5. Here the flow of the liquid cleaner, instead of being applied to the exterior as in the other examples, is applied to the interior, but the principle is the same: a large surface area to be cleaned, bath−soiling agent separation, and contact by flow.

As shown above, the BSF method can be highly useful for laboratory simulation of numerous real systems for cleaning hard surfaces. After a device is described on the basis of the method proposed, its relevance will be shown below both for the study of detergency dynamics and for the evaluation of the detersive effectiveness of detergent components, such as surfactants and dispersive agents, as well as formulations of complete detergents.

1.2. Dynamic Evaluation of Detergency. The BSF method can be used to study the dynamics of washing and to establish appropriate mathematical models. It is applicable to surfactants, to detergent formulations under development, to builders, and to complete formulations.

The elimination of grease from a substrate is a nonspontaneous process in which the addition of detergent reduces the amount of energy needed to eliminate the greasy deposit.[2] In addition, once transferred to the bath tank, the grease is distributed in finely divided droplets, which in turn can be deposited on surfaces, so that the effectiveness of the wash is determined by the balance achieved between the capacity of elimination and the redeposition of the soil. Also, from these two processes, Dimov et al.[2] mentioned the importance of taking into account the influence of the hydrodynamic conditions in the washing.

Given that the bath and the substrate are separated, it is possible to represent the flow process of the soil that passes from the substrate (S) to the bath (B), and vice versa, according to the following scheme:

$$\mathrm{S} \underset{k_2}{\overset{k_1}{\rightleftharpoons}} \mathrm{B}$$

where k_1 represents the kinetic constant of the process directed toward the bath and k_2 the kinetic constant of the reverse process. This scheme is not intended to represent a pure thermodynamic bath–redeposition equilibrium but rather the trend of the soil reaching the bath (including the effect of the detergency plus the hydrodynamic effect) and the contrary effect of the soil remaining on the substrate (including therefore the effect of the redeposition plus the hydrodynamic effect of the system). If we accept that the two opposing processes are of the first order with respect to the mass of soil both on the substrate and in the bath, the differential expression of the model proposed would be

$$\frac{\mathrm{d}m_\mathrm{B}}{\mathrm{d}t} = k_1 m_\mathrm{S} - k_2 m_\mathrm{B} \qquad (1)$$

where m_s and m_B are the masses present at each time on the substrate and in the bath, respectively. Integrating the equation above, we reach the following kinetic-model expression:

$$\mathrm{De} = \mathrm{De}_{\max}(1 - \mathrm{e}^{-(k_1+k_2)t}) \qquad (2)$$

where De is the detergency expressed as a quotient between the mass of soil remaining in the bath, on the one hand, and, on the other hand, the total mass of soil in the system, $m_\mathrm{S} + m_\mathrm{B}$. On the other hand, $\mathrm{De}_{\max}$ is the detergency that would be reached at an infinite time under the test conditions, given by the following expression:

$$\mathrm{De}_{\max} = \frac{k_1}{k_1 + k_2} \qquad (3)$$

A reordering of eq 2 gives

$$\frac{\mathrm{De}_{\max} - \mathrm{De}}{\mathrm{De}_{\max}} = \mathrm{e}^{-(k_1/\mathrm{De}_{\max})t} \qquad (4)$$

which could be linearized, and therefore a simple linear regression is possible to determine the kinetic parameters k_1 for a given $\mathrm{De}_{\max}$.

2. Experimental Section

Figure 5 offers the scheme of the device assayed for evaluating the detergency on hard surfaces by the BSF method, which is according to the scheme in Figure 1.

This device has two containers, one for the bath (450 mL in volume), 1, and the other for the packed column (2.5 cm in diameter and 8 cm in height), 2, a peristaltic pump, 3, a bath of thermostatically controlled water, 4, for maintaining a constant temperature throughout the system, and a stirrer, 5, to homogenize the bath. The total volume of the washing system is 550 mL.

The container, 1, has four orifices: in the first, a thermometer can be inserted, 17, to check the exact temperature of the wash; in the second, the tubing enters, 8, which, coming for the column, returns the bath water to the mixing tank; in the third, 5, the stirrer is inserted, to keep the bath homogenized; and, finally,

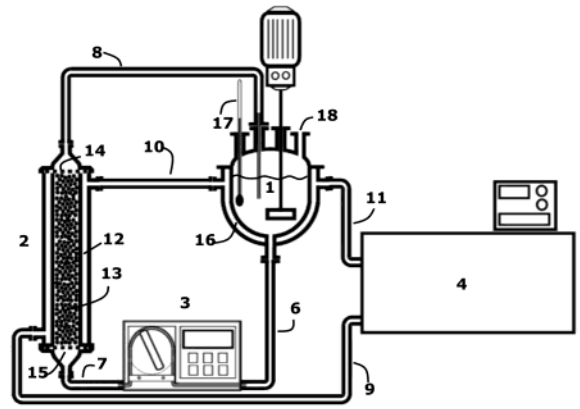

Figure 5. Experimental device using the BSF method.

in the fourth, a pipet can be connected to extract samples to be analyzed. The bath exits from the underside through tubing 6, reaching the peristaltic pump, which pumps it through 7 to the column, and it finally returns through 8, finishing the cycle. In addition, the container, which contains the bath, is jacketed, 16, to ensure a constant temperature, having an intake for the tubing 10 and an exit toward the tubing 11, through which the hot water from the thermostat-controlled bath, 4, circulates.

The packed column, 2, contains the soiled substrate, 13, in the form of glass spheres of 0.3 cm in diameter held by a perforated support in the lower part, 15, and another support in the upper part, 14. The column is also jacketed, 12, passing through the interior of the water jacket heated by the thermostatically controlled bath, 4, through the tubing 9 and exiting toward the mixing container, 1, by the tubing 10. The function of the peristaltic pump, 3, is to ensure the circulation of the bath through the system, with an adequate and carefully regulated flow.

The soiling agents used were oleic acid as a liquid soiling agent (from Panreac) and a solid grease mixture of 26% EDENOR LLSM GS from Henkel (formed by 48.4% stearic acid, 50% palmitic acid, and other components) and 74% oleic acid. The detersive agents were BEROL LFG 61 (from Azko Nobel) and CELLESH 100 and CELLESH 200 (from Kao Corp.).

The detergency was evaluated as a percentage of the soil eliminated from the substrate and was analyzed by the acidity index of the different greases tested.

The following series of experiments were programmed: experiment 1 to determine the capacity for establishing detersive models (for the parameters used, see Table 2); experiment 2 to analyze the influence of the flow rate through the packed column; experiment 3 to study the effect of the stirring in the tank containing the wash bath; experiment 4 to analyze the influence of the wash temperature; experiment 5 to design experiments that, together with the BSF method, enable the establishment of the conditions of maximum detergency for the surfactant BEROL LFG 61. The specific conditions for washing are indicated for each of the experiments.

3. Results and Discussion

The series of experiments programmed were executed to determine the effectiveness of the method proposed to assess detergency.

Table 2. Conditions of Test Wash 1

washing conditions	0.5 g/L of CELLESH 100 0.5 g/L of CELLESH 200 300 mg/L hardness as $CaCO_3$ 64.6 g of glass spheres of 0.3 cm in diameter $T = 45$ °C soil = 15 g/L type of soiling: mixture of 26% Edenor LLSM GS (formed by 48.4% stearic acid, 50% palmitic acid, and other components) and 74% oleic acid pH initial wash = 8.0 flow rate = 30 L/h volume of bath = 500 mL
detergency evaluation	acid−base titration of the quantity of fatty acids in the bath, according to the standardized technique of measuring the acidity index

Table 3. Results of the Wash for Test 1

time, t (min)	De mean (%)	standard error	time, t (min)	De mean (%)	standard error
0.00	0	0	1.00	50	1.3
0.25	30	2.5	1.25	52	1.3
0.50	45	1.3	3.00	53	2.5
0.75	48	2.5			

Table 4. Kinetic Parameters and Determination Coefficient

De_{max} (%)	50.28	k_2 (min^{-1})	2.171
k_1 (min^{-1})	2.195	R^2	0.9986

Table 5. Calculated vs Experimental Detergency Values

time, t (min)	De (%)	De calcd (%)	time, t (min)	De (%)	De calcd (%)
0	0.00	0.00	1	49.64	49.64
0.25	30.27	33.40	1.25	50.06	50.06
0.5	44.80	44.61	3	52.83	50.27
0.75	48.43	48.37			

3.1. Test 1: Capacity To Establish Detersive Models. As indicated above, the BSF method can be used to study the dynamics of washing and to establish appropriate mathematical models.

Table 2 shows the washing parameters of test 1, while Table 3 gives the results for detergency under the conditions of Table 2. This test is an example in which the detergent is formed by only two commercial dispersants of soil, the two being CELLESH 100 and CELLESH 200, of the Kao Corp., SA, without surfactants. Detergency is therefore due to the dispersant effect of these commercial builders. For each time period, three measurements were made (standard error specified in Table 3).

These values were adjusted by regression to the model expressed in eq 2 with the help of the linearizable eq 4. The values of De_{max} and the kinetic coefficient k_1 were determined (Table 4). The value of the determination coefficient was particularly good, implying excellent behavior of the proposed device, as well as the possibility of adjusting the empirical values to theoretical models.

Table 5 provides a comparison of the experimental and calculated values, showing excellent agreement.

3.2. Test 2: Influence of the Flow Rate. Mechanical action is perhaps the most determining factor in the washing process. This action can be established and altered in the BSF method, altering the rate at which the bath flows from its reservoir to the packed column containing the soiled substrate. If the device and the method are appropriate, changes in the flow rate should significantly vary the detergency.

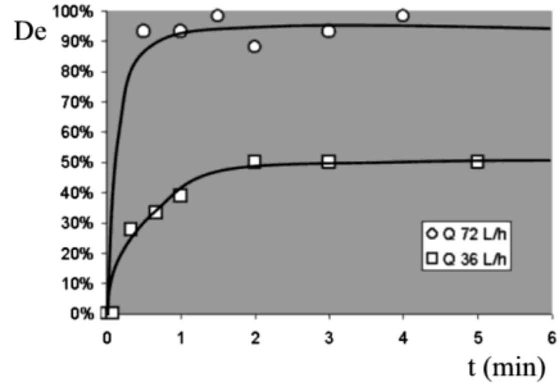

Figure 6. Influence of flow rate.

To confirm this influence, test 2 was performed. As a detergent, a commercial tensioactive mixture, BEROL LFG 61 (Akzo Nobel), was used as recommended by CIP cleaners and for dishwashing detergents. The soil was composed of oleic acid, this being determined in the bath by extraction of samples at various times and subsequent acid−base titration. The washing temperature was 40 °C, and there was practically nil calcic hardness. The test was made at two different flow rates, 36 and 72 L/h. For a bath volume of 300 mL, 17.15 g of soil was used with the lower flow rate and 16.18 g for the higher one.

Figure 6 shows clearly that a higher flow rate (stronger mechanical action) resulted in greater detergency. Twice the flow rate gave a twofold better wash. It was also verified that not only was the De_{max} altered, but this was also achieved at high flow rates before low ones (less than 1 and 2−3 min, respectively). Evidently, to conduct washing tests in which the influence of other variables can be clearly noted, it is not necessary to use low flow rates for the bath circulation.

Similarly, this test shows that the BSF method, as well as a device based on this method, is capable of adequately evaluating the influence of the mechanical action in the washing processes.

3.3. Test 3: Influence of Stirring in the Bath. In accordance with the scheme of the BSF method in Figure 1 and with the device used (Figure 5), the wash can be stirred or not. In the case of low or null stirring, it would be expected that part of the soil that reaches the recipient from the packed column is redeposited on the column walls, for which the free soil in the bath slightly declines. On the other hand, a good stirring brings about a strong mechanical action against these walls, and the soil present in the bath is hardly redeposited.

These two possibilities are highly interesting because they enable the study of two types of real cases. Little or no stirring simulates cases, for example, of floor-washing machines. On the other hand, in the case of dishwashing machines, the system with stirring is more appropriate (Figure 2). The CIP circuit cleaning systems (Figure 4), depending on whether there is good stirring or not in the mixing container, corresponds to one or the other case.

To verify this effect and the degree of sensitivity of the BSF method against this phenomenon, test 3 was performed. Washing was performed only with distilled water to avoid the anti-redeposit effect of certain detergents and detergent components that masked the phenomenon to a certain degree. The conditions were a

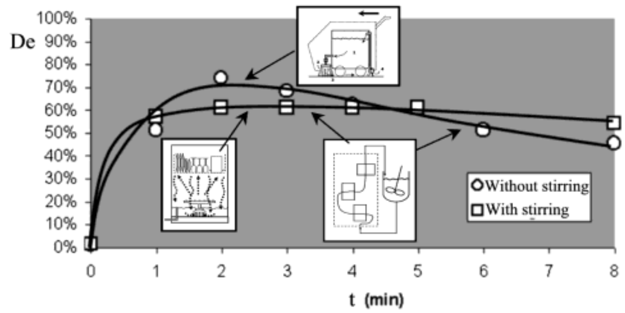

Figure 7. Influence of stirring.

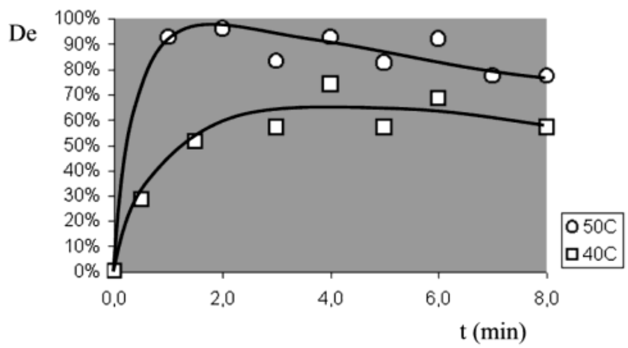

Figure 8. Influence of temperature.

temperature of 40 °C, a flow rate of 60 L/h, 17.374 g of soil, and 480 mL of bath. In one experiment, stirring was at 150 rpm and in the other 0 (nevertheless, there was always an appreciable degree of mixing because of the hydrodynamics of the system itself). Figure 7 shows the results of these two experiments. With stirring, the highest detergency values were reached and these remained approximately constant. On the other hand, without stirring, when the high was reached, the soiling values detected in the bath were progressively diminished by the effect of the redeposition on the walls of the bath tank. This test demonstrated that the BSF system, and devices based on it, can detect changes in the hydrodynamics of the wash system and therefore adapt adequately to the type of washing machine to be simulated.

3.4. Test 4: Influence of Temperature. Another basic variable in detergency is temperature. The BSF method and the devices based on it should be sensitive to small variations in temperature. In this sense, test 4 was made, where only the temperature was varied. The conditions in which the test was performed were a flow rate of 50 L/h, use of distilled water as the bath, and no stirring.

Figure 8 gives the results for 40 and 50 °C, showing detergency to be slightly higher at 50 °C. With this test, it was also confirmed that the method and the device proposed are also adequate to analyze the effect of this variable.

3.5. Multivariate Evaluation of Detergency. The detersive capacity, as demonstrated above, depends on numerous factors. For example, in the formulation of a detergent with the highest cleaning efficacy, a number of experiments must be conducted, changing all of the variables affecting the process such those analyzed above: flow rate, stirring, temperature, type and quantity of soil, type and position of the substrate, composition of the bath (concentration and type of surfactant and builders). As a means of minimizing this enormous number of experiments, the use of techniques of the central circumscribed compound (CCC) experimental design is advisable.

The experimental design by CCC consists of estimating a central point of the coordinates x_1, x_2, ..., x_n near the optimum. This point is the arithmetical mean of two extreme values to the right and left of each variable called (−1, +1) and two more distant points called (−α, +α). For each variable, this involves making five different levels −α, +1, 0, +1, and +α while choosing the combinations that fit the established geometric design. The α value depends on the number of variables (k) of the experiment, calculated from the expression

$$\alpha = (2^k)^{1/4} \tag{5}$$

with α = 1.68 for the case of using three variables (k = 3).

Therefore, for each experimental design, it is necessary to fix a set of variables and structure the matrix of corresponding experiments according to the geometric design chosen.

The results can be adjusted to a surface-response method by a second-degree polynomial expression:

$$\mathrm{De} = b_0 + \sum b_i X_i + \sum\sum b_{ij} X_i X_j + \sum b_{ii} X_i^2 + e \tag{6}$$

where De is the detersive capacity and x_i the factors or variables to be correlated with it. The expression contains a first-degree term that represents a linear relationship considered as principal, another term in which the variables cross and which represents the influence of some over others, and finally a second-degree term that refines the previous one and which enables the calculation of maximums and minimums, that is, optimal values of the dependent variable. The parameters b_0, b_i, and b_{ij} are constants, and e is an error term or residual between the observed value and the calculated one. The experimental values adjust to the previous equation by polynomial regression, and the usual statistical methods can be used to determine the goodness of fit.[10] On these premises, the BSF method can be used to perform multivariate analyses, regardless of their complexity, with a very small experimental matrix and with satisfactory results. This approach is without a doubt highly useful for formulating detergents with the optimized detersive capacity or as a means of comparing, in very unequal situations, various test or commercial detergent compositions.

3.5.1. Test 5: Determination of the Detersive Effectiveness of BEROL LFG 61. This test was performed by a CCC design of three variables, with the detergent agent being a product habitually used in CIP cleaning, BEROL LFG 61 (by Akzo Nobel), with oleic acid as the soil. The variables were[11] temperature, T (°C), a Napierian logarithm of the total soil concentration, O (g/L), and a Napierian logarithm of the detergent concentration, C (g/L). The other variables were maintained constant, with a flow rate of 45 L/h, stirring at 300 rpm, and null calcic hardness. The results for detergency are shown in Table 6.

After a polynomial regression, the detergency was adjusted to

$$\begin{aligned}\mathrm{De} = {} & -267.016 + 117.425 \,\mathrm{Ln}\, O + 61.9972 \,\mathrm{Ln}\, C + 4.5143T - 9.78087(\mathrm{Ln}\, O)^2 - 0.1097(\mathrm{Ln}\, C)^2 - \\ & 0.0379107T^2 - 17.9281 \,\mathrm{Ln}\, O \,\mathrm{Ln}\, C - 0.30942T \,\mathrm{Ln}\, C - 0.293827T \,\mathrm{Ln}\, O\end{aligned} \tag{7}$$

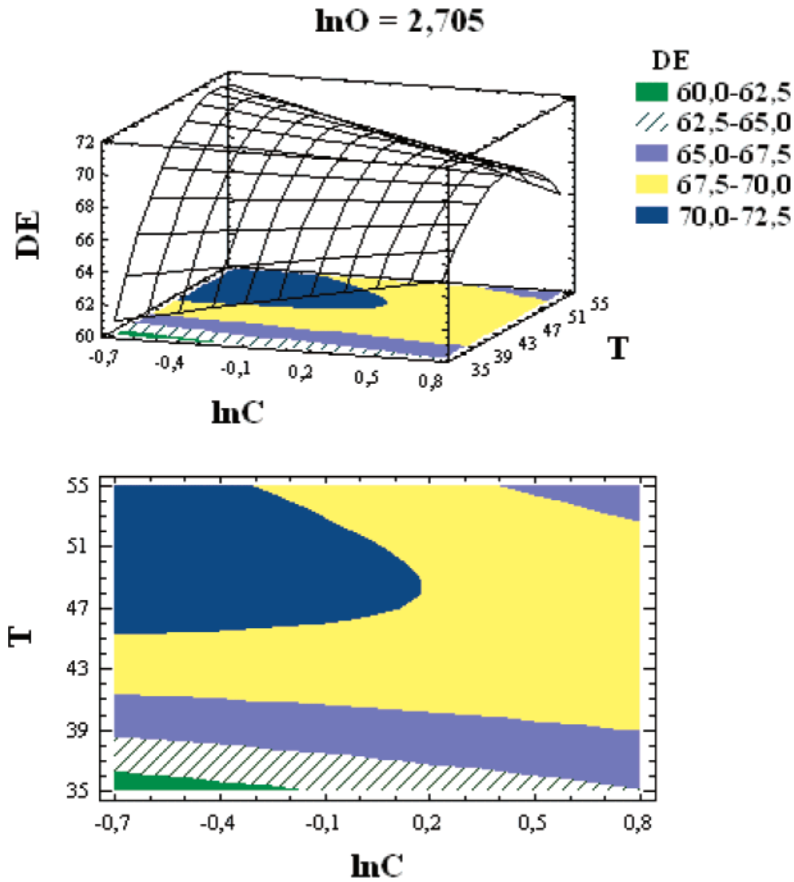

Figure 9. Example of surface response.

Table 6. CCC Design for Determining the Effectiveness of BEROL LFG 61 Detergent

	experiments with BEROL LFG61 and oleic acid										
	ooded levels			real values			transformed values			De (%)	
expt	Ln *O* (g/L)	Ln *C* (g/L)	*T* (°C)	Ln *O* (g/L)	Ln *C* (g/L)	*T* (°C)	*O* (g/L)	*C* (g/L)	*T* (°C)	obsd	calcd
1	−1.00	1.00	1.00	2.30	0.69	55.0	10.0	2.0	55.0	48.65	50.29
2	−1.00	−1.00	1.00	2.30	−0.69	55.0	10.0	0.5	55.0	47.59	45.12
3	−1.00	−1.00	−1.00	2.30	−0.69	35.0	10.0	0.5	35.0	32.08	32.32
4	1.00	−1.00	−1.00	3.11	−0.69	35.0	22.4	0.5	35.0	86.67	86.27
5	1.00	−1.00	1.00	3.11	−0.69	55.0	22.4	0.5	55.0	97.74	94.31
6	−1.00	1.00	−1.00	2.30	0.69	35.0	10.0	2.0	35.0	41.36	46.03
7	1.00	1.00	1.00	3.11	0.69	55.0	22.4	2.0	55.0	78.44	79.43
8	1.00	1.00	−1.00	3.11	0.69	35.0	22.4	2.0	35.0	76.23	79.93
9	-α	0.00	0.00	2.02	0.00	45.0	7.6	1.0	45.0	32.02	29.94
10	α	0.00	0.00	3.39	0.00	45.0	29.6	1.0	45.0	99.97	100.20
11	0.00	-α	0.00	2.70	−1.16	45.0	15.0	0.3	45.0	65.80	69.64
12	0.00	α	0.00	2.70	1.16	45.0	15.0	3.2	45.0	74.97	68.87
13	0.00	0.00	-α	2.70	0.00	28.2	15.0	1.0	28.2	58.05	53.51
14	0.00	0.00	α	2.70	0.00	61.8	15.0	1.0	61.8	61.57	63.89
15	0.00	0.00	0.00	2.70	0.00	45.0	15.0	1.0	45.0	72.38	69.40
16	0.00	0.00	0.00	2.70	0.00	45.0	15.0	1.0	45.0	70.81	69.40
17	0.00	0.00	0.00	2.70	0.00	45.0	15.0	1.0	45.0	65.49	69.40

The determination coefficient, $R^2 = 0.975$, was notably high, given that three variables were handled simultaneously. The absolute mean error was estimated at only 1.45%. Table 6 also shows the observed and calculated values of detergency, indicating good agreement.

From eq 7, it is possible now to study the importance and weight of each variable involved and to predict the behavior of BEROL LFG 61 in diverse cleaning circumstances. As an example, Figure 9 shows, for a fixed quantity of soiling agent, the detersive behavior of BEROL LFG 61 against the variation of the detergent concentration and temperature. This tensioactive mixture presents maximum efficacy at temperatures of close to 45 °C. Values above this temperature worsen the cleaning results. In addition, within the range studied, an excess of detergent does not improve, and even worsens, the detersive results.

4. Conclusions

The method called BSF and the device based on this method are valid to evaluate the detersive and dispersant effectiveness of detergents for hard surfaces. We propose, therefore, to determine the detersive efficacy of commercial detergents, as a means of comparing detergents under development in order to optimize formulations, and to correlate the detersive capacity of surfactants with their structure or physicochemical properties, and even other components usually in detergent formulas, such as dispersants (phosphates, phosphonates, acrylic and maleic polymers, nitrilotriacetic acid, ethylenediaminetetraacetic acid, etc.) or enzymes, especially proteases, lipases, and amylases. In addition, the method is appropriate to evaluate detersive kinetics.

The BSF method and the device can therefore be used with all types of cleaners that are not for textiles, and thus the field of application is very broad. Nevertheless, this is preferentially recommended for dishwashing detergents, floor washers, and tunnel washes and for cleaning circuits in the agrofood industry, as well as in all cases in which the surface to be cleaned makes contact with the bath by flow applied by spray, squirting, or other systems.

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