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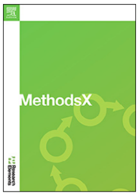

A universal method for determining the detergent effect of surfactants

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ABSTRACT

Surfactants are indispensable in industrial applications today due to their wetting, emulsifying, dispersing, cleansing, and detergent properties. The use of surfactants extends from the cosmetic industry to the petroleum industry and beyond.

Their characteristics and effectiveness can be assessed through various standardized tests, and based on these methods, their applications can be determined. However, there is a lack of a universally applicable testing method for one crucial and complex property: the detergent effect.

The detergent effect refers to the removal of unwanted contaminants from a solid surface. However, cleaning is not solely attributed to the surfactant but to the appropriate combination of various factors, whose synergistic effect reduces surface contamination. The most significant factors influencing detergent effect include the characteristics and nature of the contaminants, properties of the cleaning solution (surfactant concentration and composition, water hardness, enzymes, etc.), temperature, washing time, and hydrodynamic conditions. Additionally, the presence of electrolytes, pH of the cleaning solution, and detergent foaming properties may also play important roles.

Our goal was to develop a detergent effect testing methodology that is not specific to any particular application domain but offers a straightforward and easy-to-implement solution for comparing the detergent effect of various types of surfactants.

- The study presents a method for determining detergent effect of surfactants.
- The method is universal and suitable for the evaluation of any type of surfactant.
- The method is low-cost and easy to perform.

Specifications table

Subject area:	Chemistry
More specific subject area:	Surfactants
Name of your method:	A universal method for determining the detergent effect of surfactants
Name and reference of original method:	N.A.
Resource availability:	1. Silica plate 60 F_{254} (100 mm) 2. Circular filter paper (100 mm, 7–12 μm) 3. SN-85 base oil (GHS08 Based on CLP) 4. Non-polar dye 5. Closable TLC chamber 6. Closable Petri disk

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Background of method

The detergent effect refers to the removal of unwanted contaminants from a solid surface. However, cleaning is not solely attributed to the surfactant but rather to the appropriate combination of various factors. The synergistic effect of these factors reduces surface contamination [1].

Determining the detergent effect is still achieved through overly specific methods, providing detailed results only for certain surfactant product groups.

The Tergotometer was developed in the 1950s specifically for measuring the detergent effect of detergents. Today, it is commonly used for testing detergents as well as their modified versions for dishwashing purposes.

Measurement standards primarily define the type of contamination. The ASTM D2960 [2] standard mandates the use of "real" contaminants. However, for control tests and detergent development purposes, it is advisable to use laboratory contaminants produced from known components in known proportions, as prescribed by the ASTM D3050 [3] and ASTM D4008 [4] standards.

The results are calculated from the analytical methods of colorimetry or spectrophotometry, comparing the reflectance of contaminated and washed materials [3]. However, methods suitable for investigating the detergent effect of surfactants on cleaning solid surfaces are less developed [5-8].

Alameda et al. employed a continuous operation apparatus to determine the detergent effect [9].

The previously mentioned methods are not suitable for everyday laboratory use. Our goal was to develop a method for determining detergent effect that is simple, cost-effective, quick, and easily integrated into laboratory routine.

Method details

The determination of detergent effect consists of two main steps:

1. Thin layer chromatography
2. Circular paper chromatography.

Thin layer chromatography (TLC)

In thin-layer chromatography, an 100 mm length aluminum-backed silica gel thin layer (Silica plate 60 F_{254}) is required. The thin layer should be activated in an oven at 90 °C for 1 h before the analysis to remove any absorbed moisture during storage. SN-85 base oil is used as the mobile phase, in which 0.001 wt% of non-polar dye should be dissolved.

The surfactant to be examined should be prepared as a 1 wt% aqueous solution, which will serve as the eluent. The process involves dropping 4 µl of colored SN-85 base oil onto the thin-layer plate and then placing the plate into the chamber containing the 10 ml surfactant solution.

The experiment should be conducted using ascending chromatography in a saturated vapor chamber, so the chamber must be kept at a constant temperature of 40 °C in an oven for 3 h during the experiment.

During thin-layer chromatography measurements, the experiment is as shown in the Fig. 1.

The result of the experiment is the distance in millimeters measured from the spot where the mobile phase was applied to the mobile phase front.

Circular paper chromatography (CPC)

In the case of circular paper chromatography, a circular filter paper with a diameter of 100 mm and a pore size of 7–12 µm should be used. 4 µl of colored SN-85 base oil should be dropped onto the center of the paper as the mobile phase. Once the spot has dried, the filter paper is placed horizontally onto a Petri dish filled with a 1 wt% aqueous solution of the surfactant to be tested, ensuring that the paper wick is immersed in the solvent. Through the wick, the solvent is released, dividing the components into concentric rings. Unlike thin-layer chromatography, horizontal chromatography is used here, with the solvent moving horizontally from the point of application towards the circumference of the paper. The arrangement of the experiment is shown in Fig. 2.

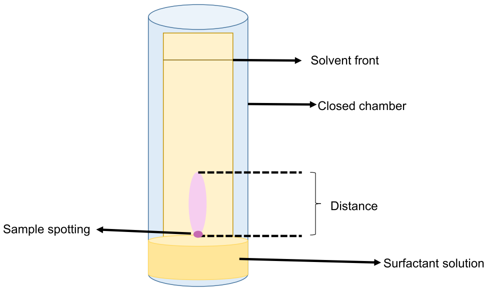

Fig. 1. Thin layer chromatography (TLC).

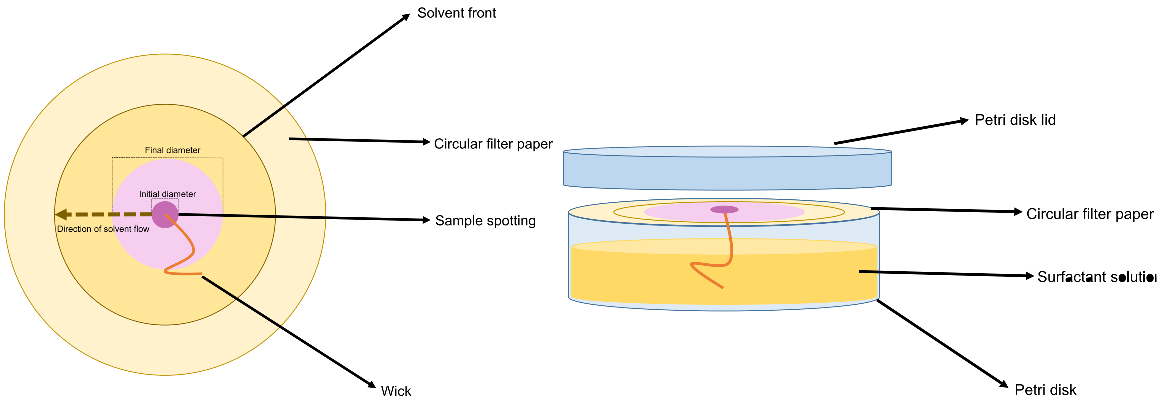

Fig. 2. Circular paper chromatography (CPC).

During circular paper chromatography measurements, the run must also be conducted for 3 h at a temperature of 40 ˚C closable Petri dish, meaning that in this case as well, the solvent creates a saturated vapor chamber.

The result of the experiment is the difference in diameter in millimeters between the original spot and the spot following chromatography.

Evaluation system

During the evaluation, it is essential to consider the differences between the two methods. Based on experience, some surfactants exhibit notable results in thin-layer chromatography, while their detergent effect obtained through circular paper chromatography is less significant. Therefore, to assess the results effectively and provide comprehensive insight into the detergent activity of surfactants, an evaluation system is applied that can appropriately represent the results. Taking these factors into account, the equation for the complex evaluation is as follows (Eq. (1)).

$$\mathrm{DE} = \frac{a+b}{100\ \mathrm{mm}} \tag{1}$$

where

DE = detergent effect

a = result obtained from thin layer chromatography, mm

b = result obtained from circular paper chromatography, mm

The maximum values of the parameters are determined by the geometric dimensions of the measuring instruments. For instance, the maximum length of the silica gel plate used in thin layer chromatography is 100 mm, while for circular paper chromatography, the maximum diameter is also 100 mm. These values define the possible range of the DH parameter, which ranges from 0 for surfactants without detergent action to a maximum of 2 for highly effective surfactants. If the surfactant reaches the limit of 2, the equation and thus the maximum limit can be modified by adjusting the measurement conditions and geometries.

Based on the DH values, surfactants can be grouped according to their detergent action. The grouping intervals are as follows:

- 0.00–0.40 – the detergent effect is non-determinable
- 0.41–0.80 – the detergent effect is low
- 0.81–1.20– the detergent effect is moderate
- 1.21–1.60 – the detergent effect is good
- 1.61–2.00 – the detergent effect is excellent.

The determination of some surfactant detergent effect has been completed. The main properties of the examined surfactants are listed in the Table 1.

The classification of the detergent effect of some surfactants is included in the Table 2.

Repeatability: The difference between the results of two consecutive measurements conducted by the same person, using the same equipment, under constant test conditions, on the same test material, should not exceed the following value in the long term, provided that the test method is executed accurately and consistently: $r = 0.1$X out of 20 cases at most. Where X is the average of the results to be compared.

Reproducibility: The difference between the results of two independent measurements performed on the same test material by different individuals in different laboratories should not exceed the following value in the long term, provided that the test method is executed regularly and accurately: $R = 0.15$X. Here, X represents the average of the compared results.

The developed method is suitable for determining the detergent effect of the surfactants presented and investigated. The method allowed for the classification of the surfactants under examination. The determined detergent effect value using the method could contribute to increasing the efficiency of the selection process for surfactants or surfactant mixtures to be used in various industrial processes.

Table 1
Main properties of the used surfactants.

Surfactant sign	S-1	S-2	S-3	S-4	S-5	S-6	S-7
Product name	Sodium dodecylbenzene sulfonate	Hostapur® SAS 60	Sodium laureth sulfate	Genapol® OA 040	Cocamide DEA	Lutensol® XA 50	Genamin® T-020
Type	anionoic	anionoic	anionoic	nonionic	nonionic	nonionic	nonionic
Manufacturer	Roth	Clariant	EMAL	Clariant	Alpha Chemicals	BASF	Clariant
Apperance	Light yellow liquid	Yellow pasta	Light yellow pasta	Light yellow liquid	Light yellow liquid	Light yellow liquid	Yellow pasta
Kinematic viscosity at 40 °C, mm/s^2	1014	–	–				
Dynamic viscosity at 40 °C, mPas	978	–	–				
HLB	11.7	11.7	40	10.8	12.1	11.5	12.1
Transmittance in water (1 wt%)	96	100	100	9	21	21	5

Table 2
Classification of the detergent effect of some surfactants.

Surfactant sign		S-1	S-2	S-3	S-4	S-5	S-6	S-7
TLC, mm		15	28	6	24	40	70	1
CPC	Initial diameter,mm	15	19	18	19	18	17	17
	Final diameter,mm	41	45	58	42	65	83	25
	Difference, mm	26	26	40	23	47	66	8
DH value		0.41	0.54	0.46	0.47	0.87	1.36	0.09
Qualification		Low	Low	Low	Low	Moderate	Good	Non-determinable

Safety precautions

- SN 85 base oil may be fatal if swallowed and enters airways (H304).
- If medical advice is needed, have the product container or label at hand (P101).
- If swallowed, immediately call a Poison Center or doctor/physician. Do NOT induce vomiting (P301+P310+P331).
- Store locked up (P405).
- Dispose of contents/container to an approved waste disposal plant (P501).

Ethics statements

None.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Rebeka Bejczi: Writing – original draft, Formal analysis. **Roland Nagy:** Conceptualization. **Anna Molnár-Kiss:** Methodology.

Data availability

No data was used for the research described in the article.

Acknowledgments

None.

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