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CLEANING UP THE COOKSHOP

Performance of low surface energy finishes evaluated for food sector. By Heidi Van den Rul and Joey Bosmans, Sirris Smart Coating Application Lab.

Although they have obvious advantages, easy-to-clean coatings are not widely used in the food industry. Existing easy-to-clean coatings were tested for potential use in the food sector, and their potential value and shortcomings are described.

Easy-to-clean coatings have drawn attention for some decades now [1]. Many applications can be considered where a surface that can be more easily cleaned or needs less cleaning is an advantage. In the food processing industry, keeping equipment clean is a constant challenge. Control of all contamination sources requires efforts to guarantee food quality and safety. Mineral products such as $CaCO_3$, organic soils (e.g. lipids, proteins, sugars) and microbiological contaminants (bacteria, viruses, moulds, yeasts) foul the equipment [2], and can only be removed by frequently shutting down production and cleaning.

Cleaning to remove foulants has been reported to account for up to 15% of the total production time in dairy processing [3]. Easy-to-clean coatings can be a valuable part of a hygienic plan in a food factory. However, to date, coatings in general and easy-to-clean coatings specifically are not widely applied in the food industry.

The only coatings found currently on food equipment are based on fluoropolymers (PTFE) or are applied by vacuum techniques (PVD TiAlN (Titanium Aluminium Nitride), plasma coatings), mainly to reduce friction and wear. These coatings often require difficult and expensive application processes, which are impractical for larger units and for existing food processing equipment.

ANTI-FOULING COATINGS FOR FOOD APPLICATIONS

The question arises as to whether newly developed easy-to-clean coatings have potential for use in food-related applications. Significant research interest in anti-fouling materials exists, focusing on manipulation of surface properties such as wettability and topography [3, 4].

Low surface energy coatings are already commercially available on a large scale. Therefore, this study focusses on the class of 'easy-to-clean' coatings and examines their potential use in food environments.

Because cost and ease of application, especially to existing installations, are important, the search is limited to coatings that can be easily applied, e.g. by spraying. Apart from their technical features, lifetime expectancy and cost, the introduction of easy-to-clean coatings in the food sector is of course challenged by regulatory compliance. Lack of food contact approval is one obvious reason why easy-to-clean coatings are not yet widely applied in the food sector (apart from fluoropolymers that are food-approved). Of the commercial easy-to-clean coatings selected in this study (see

RESULTS AT A GLANCE

➞ Coatings are not widely applied in the food sector, although current commercially available coatings can provide easier-to-clean surfaces.

➞ Ten hydrophobic and superhydrophobic coatings were evaluated for food processing use. Although there is some correlation, the coatings showing the best contact and sliding angles are not always the best in easy-to-clean tests.

➞ Abrasion resistance can also influence the choice of coating. Different coatings provided the best results over the two substrates tested.

➞ Easy-to-clean tests are thus always recommended to complement traditional contact and sliding angle analyses. However, general standardised easy-to-clean tests do not exist.

➞ If food contact approval could be obtained, easy-to-clean coatings have a clear potential value in the food industry.

Table 1) only one is approved for food contact (HC3). The chosen coatings are commercially available products, with proven 'anti-dirt' properties in various applications, based on different materials and exhibiting decreased surface energy. Of the superhydrophobic coatings, the more durable ones (as indicated by the suppliers) have been chosen in this study. Two different substrates frequently used in food processing equipment were examined: stainless steel SS304 and polycarbonate PC ("Lexan PC9030"), with a coating thickness as indicated by the supplier. The data for PC will be discussed but not shown below.

WETTING BEHAVIOUR FIRST CHARACTERISED BY CONTACT ANGLE

The static contact angle of droplets of water and hexadecane (as a model substance for greasy dirt [1]) were first measured to assess the coatings' hydro- and oleophobicity (*Figure 1*). After coating, both substrates show increased water contact angles, with little difference between the two substrates. Since the water contact angles of the coatings are seen to be above 90°, all coatings can be called hydrophobic. Moreover, the superhydrophobic coatings are seen to have contact angles above 150°. On the other hand, the oleophobicity of the substrates only increased with coatings HC1-5 and SHC5.
For coating HC4, based on perfluoropolyethers with very low surface energy, oleophobic properties are to be expected. The other coatings demonstrating high hexadecane contact angles (HC3 and SHC5) can be assumed to also contain some level of fluorinated molecules. SHC5 especially shows a very high hexadecane contact angle (> 150°). The other superhydrophobic coatings SHC1 - 4 are completely wetted by hexadecane (contact angle 0°) and appear to have no oleophobic properties.

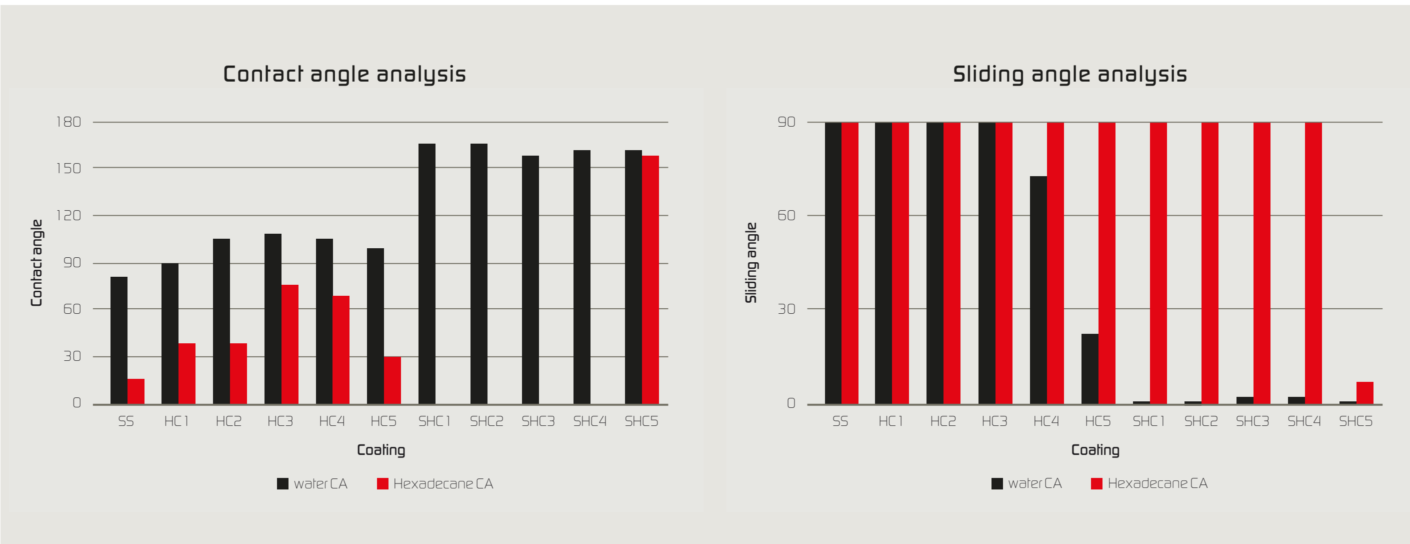
SLIDING ANGLE TEST REVEALS FURTHER DIFFERENCES

Although easy-to-clean properties are usually evaluated by static contact angle of fluids alone, the sliding angle is an additional indicator of the coating's easy-to-clean behaviour, with more value in practical conditions [1, 5]. The sliding angle of a droplet of fluid is measured by tilting the coated substrate from its horizontal position until the droplet starts to slide. The sliding angle thus measures how easy or difficult it is for droplets to stick to the surface. Using a Dataphysics "OCA15PRO" contact angle measurement apparatus, the average and standard deviation of measurements at five different positions on the coating were found. The hydrophobic coatings do not demonstrate good sliding behaviour for water droplets (*Figure 1*): a sliding angle of 90° means the droplets stick to the surface even when the substrate is tilted to 90°.
Only HC4 and HC5 show some sliding behaviour for water. The super-hydrophobic coatings, on the other hand, do what they are designed to do and show easily sliding water droplets. For hexadecane, only coating SHC5 shows sliding behaviour; its hexadecane sliding angle is even below 10°, which defines SHC5 as a superoleophobic coating.
Based on contact and sliding angle analyses, coatings HC3, HC4, HC5 and all the superhydrophobic ones are expected to be the best coatings. But how do they perform in practical easy-to-clean tests?

STAINING TEST USES THREE-STEP REMOVAL PROCEDURE

General industrial standards to objectively assess the easy-to-clean behaviour of a coating do not exist. Therefore, for each application, specific easy-to-clean tests have been developed to compare various coatings and do accelerated testing. For the evaluation of the coatings in this study, a test, containing elements extracted ➜

Figure 1: Static contact angles and sliding angles of water and hexadecane on the selected coatings applied on stainless steel.



from different standards (ASTM D1308, ASTM D4828 and ISO 10545) was employed.

The coatings are stained by nine contaminating products: liquids are applied by a pipette; products such as ketchup are smeared out across about the same size area. The staining products are selected as representative for food items and frequently seen in other tests. After drying for 24 hours at room temperature, the stains are manually removed (i) with a dry cloth, (ii) if the stain is not removed, using a wet cloth, and (iii) if the stain is still not removed, using a cloth doped in 2.5 g/l cleaning solution (sodium dodecyl benzene sulfonate, SDBS) (*Figure 2*).

After each step, a visual inspection is done, and a ranking is given to the coating: 3 if the stain is removed by dry wiping, 2 by wet wiping, 1 by SDBS wiping and 0 if the stain is not removable by this procedure. The higher the grade, the easier-to-clean the coating can be considered.

STAINING TESTS SHOW MIXED RESULTS

The results show that the coatings behave very differently (*Figure 3*). For every contaminant, one can find a coating from which the stain is removed more easily than from the substrate. But of all coatings, only hydrophobic coatings HC1, 3 and 4 behave better than or equal to the uncoated substrate SS for all contaminants tested. Of the superhydrophobic coatings, none results in a surface easier to clean than the substrate for all contaminants, but where they can repel the contaminant, they almost always score the highest grade. Also remarkable is that the superhydrophobic coatings are not solvent resistant and are always marked by MEK. Looking at the 'total' easy-to-clean value (sum of all grades and height of the columns in *Figure 3*) for the different coatings, HC1 (polyurethane), HC3 (hybrid silica-based food approved coating), HC4 (perfluoropolyether coating) and SHC2 and SHC5 (superhydrophobic coatings) have the best properties on the SS substrate. On PC, only HC1 and HC4 are slightly better. The contact angle and sliding angle analysis earlier showed that HC3, HC4 and HC5 and all the superhydrophobic coatings were to be preferred, but the easy-to-clean test can make a further distinction between the coatings. Moreover, while the polyurethane coating HC1 does not demonstrate a high water or hexadecane contact or sliding angle, it appears to be a good easy-to-clean coating in the staining test.

ABRASION TESTS ASSESS DURABILITY

In an actual application, the lifetime expectancy of an easy-to-clean coating is largely determined by its resistance to abrasion. The micro/nanostructure of superhydrophobic coatings is known to be easily damaged by

Table 1: Description of the easy-to-clean coatings tested.

	Properties	Composition	Application	Curing	Thickness (µm)
		Hydrophobic coatings			
HC1	Transparent, scratch resistant, dense	Polyurethane-based	Flow	UV	1
HC2	Transparent, anti-graffiti	Polysilazane-based	Flow	RT	30
HC3	Transparent, antibacterial, hydrophobic, HACCP approved	Hybrid nanocoating based on SiO_2	Spray (by supplier)	RT	/
HC4	Transparent, highly repellent to water and oils, mechanically stable	Functionalised perfluoropolyethers	Spray (by supplier)	Thermal	3
HC5	Transparent, superior water repellency, scratch resistant, hard	Nanostructured hybrid coating	Spray (by supplier)	Thermal	10
Superhydrophobic coatings					
SHC1	More durable, more permanent		Spray	130 °C	14
SHC2	Durable, extra top layer to enhance superhydrophobic effect		Spray	RT	12
SHC3	Greater abrasion resistance than existing superhydrophobic coatings		Spray	120 °C	/
SHC4	Superhydrophobic, durable		Spray	RT	16
SHC5	Superhydrophobic and oleophobic, extended lifetime		Spray	RT	45

Table 2: Summary of the best performing coatings in the different tests.

Best coatings		HC 1	HC 2	HC 3	HC 4	HC 5	SHC 1	SHC 2	SHC 3	SHC 4	SHC 5
Based on contact angle	water			■	■		■	■	■	■	■
	hexadecane			■	■						■
Based on sliding angle	water					■	■	■	■	■	■
	hexadecane										■
Based on easy-to-clean test	on SS	■		■	■			■			■
	on PC	■			■						
Based on abrasion resistance	on SS			■	■						■
	on PC				■	■					■

soft abrasion, e.g. during cleaning, resulting in loss of easy-to-clean behaviour. A soft abrasion test was therefore applied on the coatings, in order to reveal differences between them. The procedure uses a Taber Linear Abrasion Tester equipped with a Crockmeter kit accessory. During the abrasion, a cotton cloth is moved over the substrate back and forth (= 1 cycle) with a top weight of about 420 g. The water and hexadecane contact angles and sliding angles are then re-measured after a number of cycles.
During the test (*Figure 4*), the hydrophobicity of coatings HC1, 2 and 5 on stainless steel is seen to decrease rapidly (in less than 100 cycles) with a contact angle value that becomes lower than 90°. The reason for the rapid degradation of HC1 probably has to do with its lower thickness (1 µm compared to 10-30 µm for the other coatings). However, despite its low thickness of 3 µm, the perfluoropolyether coating HC4 remains hydrophobic up to 500 cycles of testing. HC3, the silica-based hybrid coating, has the highest abrasion resistance and maintains its hydrophobic character until the end of the test (1000 cycles). For the plastic substrate (data not shown), HC4 and 5 are the most durable and HC3 is rapidly degraded, indicating it is not suited for application on polycarbonate.
The superhydrophobic coatings in general are much more sensitive to abrasion (*Figure 4*): SHC1, 2, 3 on SS rapidly lose their superhydrophobic properties during the Crock test (water contact angle decreases to < 150° after only 20 cycles). SHC5 is the best coating regarding abrasion resistance as analysed by the Crock test and is able to maintain its superhydrophobicity until at least 40 cycles (both for SS and PC).
After 40 cycles of Crock testing, SHC5 is seen to show higher water sliding angles again (data not shown). SHC5 loses its superoleophobic properties even after five cycles. The better abrasion resistance of SHC5 might have to do with its higher thickness (45 µm) compared to the other superhydrophobic coatings (around 15 µm thickness).

Figure 2: The staining and cleaning 'easy-to-clean' test for stainless steel substrate uncoated and with coating HC3.

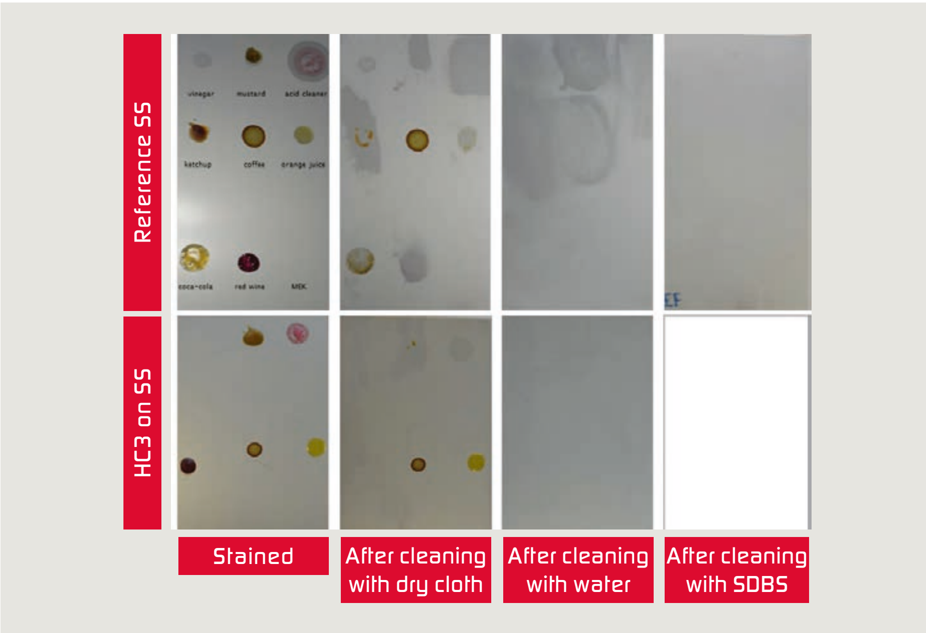
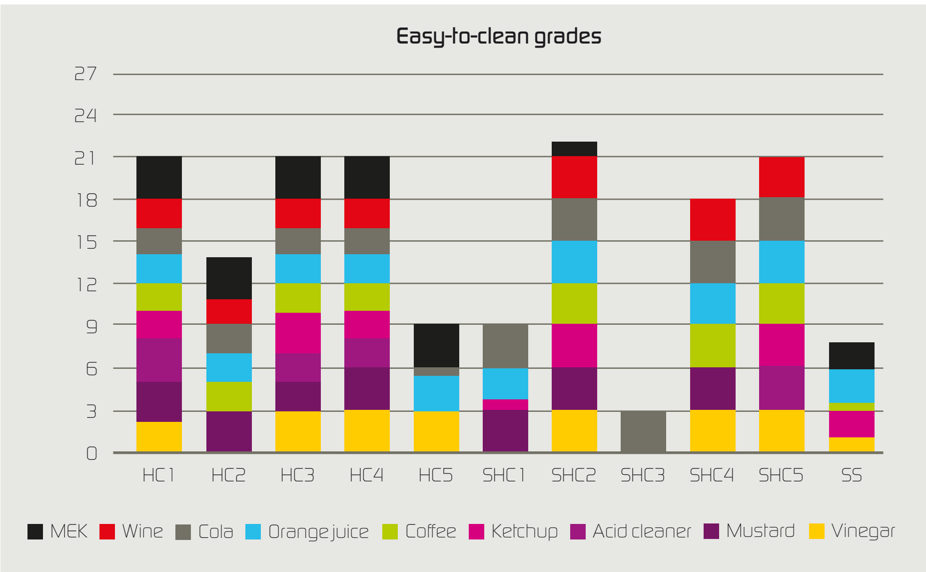


Figure 3: Grades given to each coating applied on stainless steel using the easy-to-clean test procedure for the different contaminants (highest is best).



CHOOSING THE BEST COATING IS A COMPLEX TASK

Based on all the tests, for easy-to-clean coatings in food processing industry, the best hydrophobic coating is HC3 (hybrid silica coating) or HC4 (perfluoropolyether coating) and SHC5 for the superhydrophobic ones; see *Table 2*. Although HC3 does not provide good sliding properties of liquids on top of the coating, it results in the highest water and hexadecane contact angles and the best easy-to-clean properties on SS. Also its abrasion resistance is best on SS. However, for application on plastic PC, HC4 would be the coating of choice. When a superhydrophobic coating is to be chosen, SHC5 would be the best choice, keeping in mind that its abrasion resistance is much lower than for the hydrophobic coatings.
This study shows that there is no 'best' easy-to-clean coating suitable for all situations. The composition, the substrate on which the coating is applied and the contamination it needs to repel are factors determining its behaviour. Superhydrophobic coatings do not always result in better easy-to-clean behaviour for a specific contamination; a hydrophobic coating might be a better option. Contact angle and sliding angle analyses, frequently performed as the only tests for easy-to-clean properties, are seen not to be sufficient, and should be complemented with easy-to-clean tests as close to the real application as possible.
The abrasion resistance of a coating might also determine whether it can be used in a practical situation or not. In general, superhydrophobic coatings are less abrasion resistant than hydrophobic coatings.
Taking into account all these factors, it can be concluded that there is potential for easy-to-clean coatings to be applied in the food pro-

“The food sector could also benefit from other functional coatings.“

Heidi Van den Rul
Program Manager
Sirris Smart Coating Application Lab.
heidi.vandenrul@sirris.be

3 questions to Heidi Van den Rul

Are there any other types of smart coatings for which you see potential to use in the food sector? *Apart from easy-to-clean coatings, the food sector could also benefit from other functional coatings. Scratch and abrasion resistant coatings can improve the durability of processing equipment. Hygiene is increased by using anti-bacterial coatings. Anti-fog coatings can e.g. reduce the failure of lens inspecting systems. Smart labels can protect safety and quality of food, etc. Moreover, all these functions are required in one multifunctional coating.*

What are the main requirements for coatings to obtain food approval? *Most of the regulations in Europe and the US are based on the principle that "no food contact materials shall transfer constituents into food at levels that endanger human health". In the EU there are specific regulations, but not all coating ingredients are covered. In the US, there is a difference between constituents marked as food additives that need FDA approval before use and substances that do not migrate to food. it is the responsibility of the coating manufacturer to demonstrate safety of these substances prior to marketing food contact coatings.*

As there is no standard testing for easy-to-clean-coatings what did you find important when choosing the testing methods? *Important when testing the coating is that the test simulates the actual environment of use of the coating. Parametres to consider are the staining and cleaning materials and methods, the expected abrasion level, etc.*

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cessing industry, if they are used in the right conditions and if food contact approval can be obtained.

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Figure 4: Water contact angles measured on the coatings after abrasion.