

New routes to cleanliness

Nanocomposite coatings offer advantages over PTFE in food production

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A new class of anti-adhesive coatings has been studied for potential use in industrial food production. They can be sprayed and cured at lower temperatures than current PTFE coatings. The polymer binder matrix incorporates perfluorinated comonomers and nanoscale hard particles. Polyurethane-based systems gave the best abrasion and chemical resistance, but a polyimide-based system performed well in dry processing tests.

Anti-adhesive coatings are of considerable importance for many industrial processes such as pigment and paint production as well as the food processing industry, because they can help to significantly reduce the work of cleaning. In consequence, the amount of cleaning chemicals and waste water can be reduced, which should positively affect process costs.

In this investigation, abrasion-resistant low surface free energy coatings were synthesised, based on polymer matrices containing SiC particles and copolymerised with a reactive fluoroalkyl additive to reduce surface free energy. In food production processes in particular, coating materials require high chemical stability to withstand the cleaning procedures which are used to maintain hygienic standards in the production facilities. For many decades, coatings based on polytetrafluorethylene (PTFE) and its derivatives have been used to create anti-adhesive coatings for surfaces which are in contact with food such as kitchenware and pans [1, 2].

In order to obtain long-lasting coatings of this type, intensive pre-treatment and the use of primers are necessary to obtain adequate adhesion to the substrate. The application is followed by a thermal treatment of the surface at temperatures in the range of 400 °C, which limits the types of substrate materials and requires an industrial application process.

For anti-adhesive coatings in the food production industry, it is important to provide materials which can be applied on site to existing equipment and show adhesion to different kinds of substrate materials such as stainless steel, plastics and even sometimes glass.

Nanocomposites and fluorochemicals show promise

Starting from the early nineties, both thin anti-adhesive coatings based on the sol-gel process and fluorosilanes have been investigated intensively. These combined excellent adhesion to almost all types of substrate materials with transparency and excellent hydrophobic and oleophobic properties [3].

Despite their interesting basic properties, these types of materials ultimately could not be used in food production processes because they showed low resistance to mechanical abrasion and high pH cleaning substances such as sodium hydroxide solution at elevated temperatures. Some years ago, a new class of organic-inorganic nanocomposite materials was developed that combines high abrasion stability with alkaline resistance and paintability [4]. In particular, polyimide [5] and polyurethane types [6] based on this approach have been investigated in detail in respect of their abrasion resistance, combined with heat and chemical resistance.

The polyimides in particular provide many variations with regard to monomer composition, so that flexibility, polarity and chemical stability can be varied [7-15]. In order to reduce the water uptake, many researchers have incorporated hydrophobic components, such as fluorine-containing hydrocarbons [16-23].

The approach indicated above [4, 5] mainly described the influence of the type of nanoparticulate filler on the mechanical, thermal and chemical properties. It was therefore of great interest to investigate the influence of monomer composition and fluorocarbon content of these types of polymer nanocomposite systems on the anti-adhesive behaviour under technical conditions.

The use of different types of matrices would enable appropriate coatings to be developed for very specific applications. Thus, low surface energy coatings based on the fluoroalkyl group and polymer matrices containing SiC particles were synthesised and examined for possible applications.

Formulation and production details

The nanocomposite matrices based on epoxy systems were derived from the bisphenol-A epoxy oligomer "L20" (Huntsman) in combination with "Huntsman 3261" amine type hardener. Polyurethane-based nanocomposites were derived from "Desmophen A870 BA"



Figure 1: Experimental set-up for determination of chemical resistance of nanocomposite coatings: small pots filled with test liquid were fixed on the coated substrates and placed in a convection oven at 90 °C

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Figure 2: Scheme of the morphology of the intended polymer-based anti-adhesive nanocomposite coatings (no fluorine gradient between substrate and free surface)

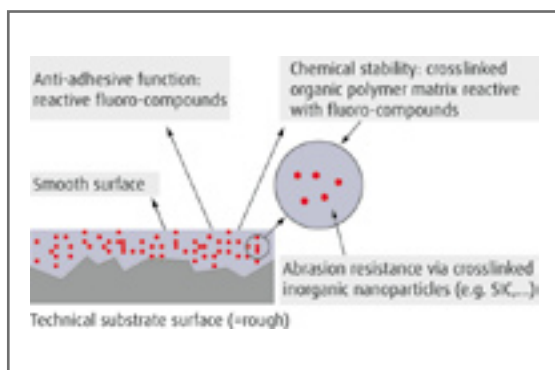


Figure 3: Result of the "Taber Abraser" test on PTFE and nanocomposite coated stainless steel after 1000 cycles

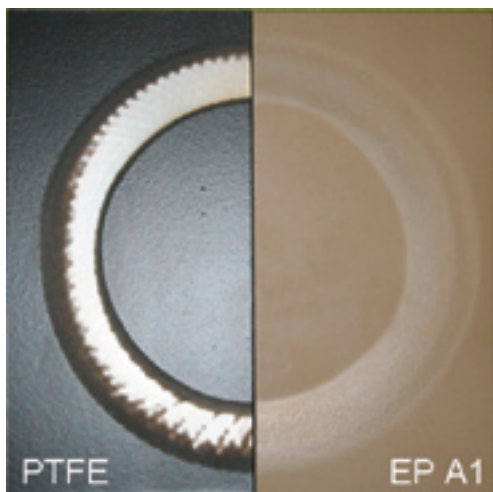


Figure 4: Direct comparison of shape of water droplet on unloaded coating surface and mechanically stressed surface (EP A1) shows that low wetting is retained after mechanical abrasion

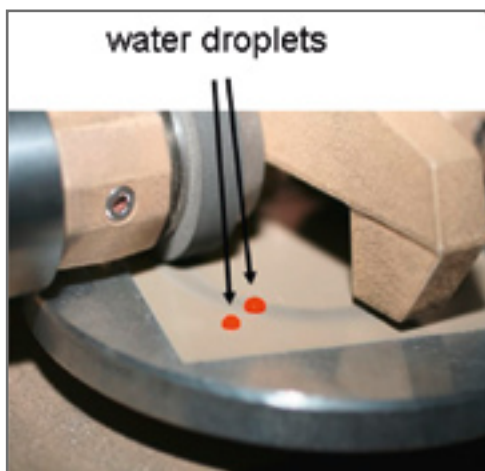


Table 1: After "Taber Abraser" test results for different materials in comparison to stainless steel (denoted ES) and polytetrafluoroethylene (PTFE) coated stainless steel; EP: epoxy matrix; PU: polyurethane matrix; PI: polyimide matrix; HD = hexadecane

Surface type	R_a [μm]	R_z [μm]	R_{max} [μm]	Weight loss after Taber test [mg]	Initial contact angle (water / HD) [$^\circ$]	Contact angle (water / HD) after Taber test [$^\circ$]
ES	0.14±0.02	0.93±0.14	1.06±0.08	-	-	-
EP A1	0.62±0.11	3.36±0.58	4.04±0.64	7.1	101 / 65	91 / 64
EP A2	1.04±0.10	4.74±0.39	6.54±0.80	5.4	100 / 64	93 / 63
PU A9	0.19±0.08	0.82±0.34	1.20±0.54	14.0	103 / 64	101 / 58
PU A67	0.25±0.07	1.33±0.42	2.33±1.25	18.0	107 / 65	104 / 63
PI A5	0.26±0.07	1.63±0.32	2.27±0.85	11.8	106 / 66	85 / 53
PI A29	0.44±0.13	2.72±0.62	3.35±0.68	4.8	101 / 68	86 / 53
PTFE	1.35±0.07	9.71±0.54	12.20±1.28	26.9	118 / 55	98 / 38

(Bayer) as polyol component and a mixture of "Desmodur N3390BA/SN" and "Desmodur Z4470BA" (Bayer) as crosslinking agents.

For all polyimide coating compositions, pyromellitic dianhydride and bis-(4-[3-aminophenoxy]phenyl)sulfone (both from ABCR) were used as matrix building monomers. All formulations also included SiC ceramic powder filler (grade "UF 10" from H.C. Starck with average particle size $d_{50} = 0.70 \mu\text{m}$) and the reactive fluoro compound "Fluorolink D" (Solvay Solexis, average equivalent weight 1000).

For the preparation of coating materials the monomers, SiC and "Fluorolink D" were mixed using a "Dispermat" bead mill in NMP as solvent. For this purpose the starting chemicals were filled together with NMP and glass beads into the milling container and were stirred with the appropriate milling tool for 2 hours at 60 °C and 1100 rpm. Then the coating material was separated from the beads by filtration to obtain a mixture ready for application.

Application and test procedures

All coating compositions were applied on stainless steel substrates (of the grade known as 1.4301 or 304, dimensions 100x100 mm) using the spray-coating method followed by thermal curing for 1 h at 130 °C for epoxy and polyurethane matrices and 2 h at 200 °C for polyimide matrices.

The determination of static contact angles was performed according to the horizontal drop method using a goniometer. For this purpose, drops of either water or hexadecane with a defined size (about 3 μl) were applied and the angle between the baseline of the drop and the tangent at the drop boundary was measured. The measurement was performed at room temperature (approx. 20 °C) and three drops per sample and test liquid were measured.

The determination of surface roughness was carried out by "Perthometer", where the surface to be characterised was scanned with a diamond stylus (scan length: 5.0 mm, scan speed: 0.1 mm/s, stylus force: 15 mg). The different roughness values R_a , R_z and R_{max} were automatically calculated from the measured surface data.

The verification of mechanical stability was performed by a "Taber Abraser" test according to DIN 53 754 (1000 cycles, 2x 500 g, CS-17 abrasive wheels) in combination with weight loss determination. To further quantify the

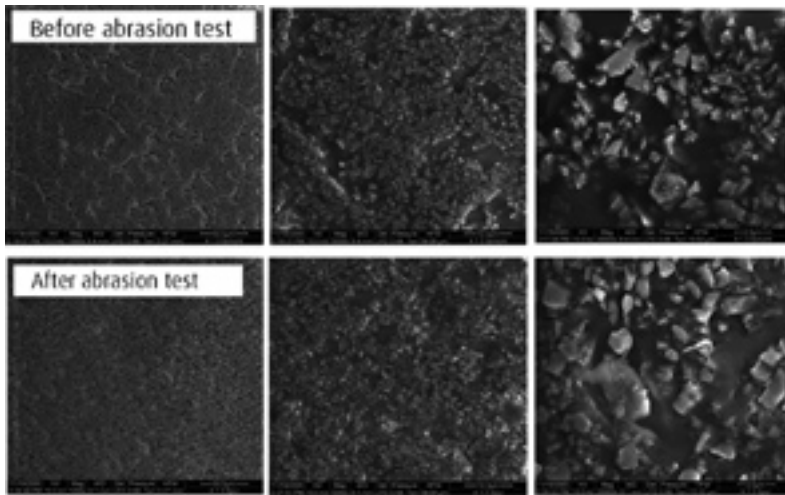


Figure 5: Scanning electron micrographs on the Taber track before and after abrasion test for epoxy-based nanocomposite system EP A1, scale bars (left to right): 50 μm, 10 μm, 2 μm

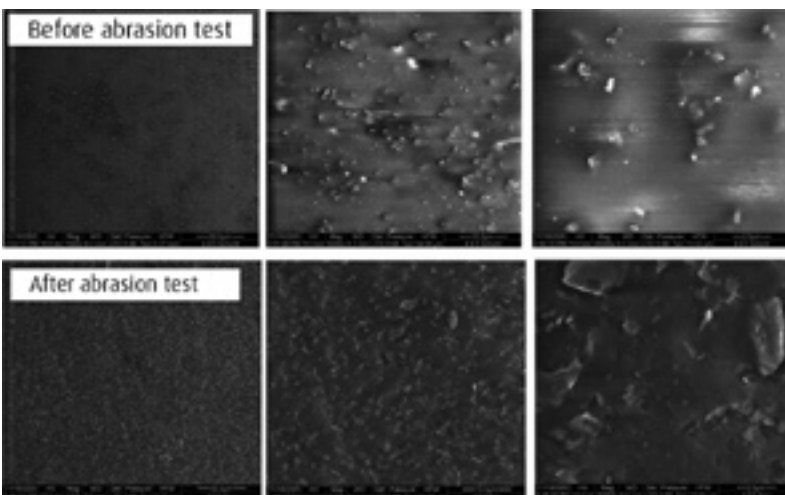


Figure 6: Scanning electron micrographs on the Taber track before and after abrasion test for polyurethane nanocomposite system PU A9, scale bars (left to right): 50 μm, 10 μm, 2 μm

Table 2: Thermal stability of wetting behaviour under dry conditions in relation to composition and exposure temperature for different nanocomposite coatings in comparison to PTFE-coated stainless steel; EP: epoxy matrix; PU: polyurethane matrix; PI: polyimide matrix; HD = hexadecane

System	Initial contact angle water/HD [°]	Contact angle water/HD after 120 h @120 °C [°]	Contact angle water/HD after 120 h @ 150 °C [°]
EP A1	101 / 65	117 / 68	133 / 76
EP A2	100 / 64	105 / 65	116 / 69
PU A9	103 / 64	109 / 68	101 / 64
PU A67	107 / 65	110 / 66	108 / 66
PI A5	106 / 66	112 / 68	103 / 61
PI A29	101 / 68	111 / 68	107 / 68
PTFE	118 / 55	118 / 57	121 / 54

mechanical stability of the different coating compositions, the static contact angles against water and hexadecane were measured by goniometer before and after this test.

In order to determine thermal stability, the coated substrates were exposed in a convection oven at either 120 °C or 150 °C for 120 hours. After cooling down at ambient temperature (approx. 20 °C) the static contact angles against water and hexadecane were again measured by goniometer to quantify the thermal stability.

In order to verify the chemical resistance, the various test liquids (NaOH (2%), HNO₃ (2%), milk (UHT milk) and carrot juice) were filled into small pots which were fixed on the coated substrates (Figure 1). Afterwards the prepared samples were put in a convection oven and were exposed at 90 °C and inspected every day. After cooling down at ambient temperature, the static contact angles against water and hexadecane were measured by goniometer to quantify the chemical stability.

Raw materials chosen to match potential applications

Different types of polymeric matrices were selected to solve different deposition and fouling problems on the surfaces of machinery in the food production industry. Because of their intrinsic hydrolytic stability, epoxy and urethane-based matrices were selected for applications in wet food processing such as thermal treatment of milk or fruit juices, whereas polyimide-based matrices were investigated in relation to dry processing.

In order to achieve the required anti-adhesive behaviour, a reactive perfluoropolyether was copolymerised and in this way introduced into the covalent polymer network. It was of interest to investigate the influence of the type of polymer on the initial hydrophobic and oleophobic properties and the stability of the anti-adhesive effect in relation to the type of external stress such as mechanical abrasion or chemical attack.

The abrasion resistance could be achieved by dispersing inorganic hard particles such as SiO₂, Al₂O₃ or SiC within the matrix. For the present investigation SiC with a particle size of $d_{50} = 700$ nm was used as a nanoparticulate filler at a constant concentration of 40 wt.%. SiC was selected because of its high intrinsic hardness (Martens hardness = 2500 MPa) and favourable electrical properties. These are relevant in order to create base systems that can provide appropriate anti-static properties in the case of dry food processing.

The nanoscale hard particles were surface-modified prior to use in such a way that nucleophilic surface groupings were established which could react with the crosslinker of the matrix material. For this reason, after thermal curing an intensive crosslinking could be achieved between the polymer matrix and the particles responsible for improving the abrasion resistance of the coating materials.

Good smoothness and abrasion resistance achieved

Figure 2 shows the morphology of these nanocomposite coatings. The standard dry film thickness of the cured layers was in the range of 15 μm to 20 μm. Table 1 shows

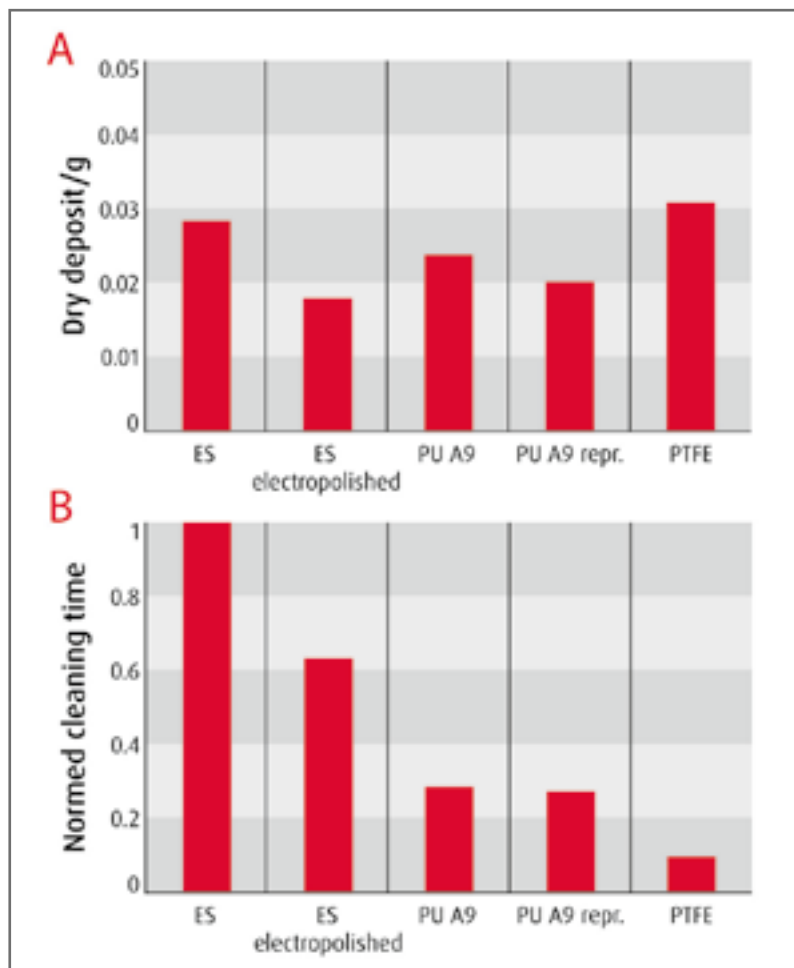


Figure 7: Left: dry deposit weight of carrot juice after treatment on heat exchanger at a surface temperature of 115 °C; right: cleaning time relative to uncoated stainless steel using swelling with 2 % NaOH then mechanical removal by rubber spatula

the evolution of the surface roughness for the different coating systems under investigation in comparison to uncoated stainless steel, together with the results from the abrasion test.

As can be observed from *Table 1*, the nanocomposite coatings show only a slight increase in surface roughness compared to the two types of stainless steel. The PTFE coated surface, used as a reference point to show the state of the art, yielded a much higher surface roughness in the micrometre range.

The abrasion test indicates a very low abrasion (weight loss) for the nanocomposite systems compared to the PTFE coated surface. After the abrasion test the PTFE coated surface was almost completely abraded, as can also be seen from *Figure 3*. The initial contact angles indicate that hydrophobic and oleophobic properties were obtained in all compositions. The weight loss of all nanocomposite coatings is quite low compared to PTFE.

It is interesting to note that after abrasion the contact angles against water and hexadecane decrease only slightly for all nanocomposites compared to their initial state. The coatings with a polyurethane matrix in particular reveal almost no change in contact angle after mechanical stress, even though their weight loss is slightly higher.

The example of the epoxy-based nanocomposite coating in *Figure 3* shows that there is only a slight track visible after abrasion testing, which is in good correspondence with the measured weight loss. The direct comparison of wetting behaviour is also shown in *Figure 4* for a water droplet which has been put on the unloaded coating surface and on the abraded track from the abrasion tester.

Epoxy and polyurethane respond differently to abrasion

As can be deduced from *Figure 4* and *Table 1*, the wetting behaviour does not change significantly due to the mechanical abrasion, which indicates that the nanocomposite coating materials have the potential to be useful for heavy-duty applications. Nevertheless it was of interest to investigate the abrasion mechanism more closely.

In this context *Figures 5* and *6* show scanning electron micrographs from the surface in the area of the abrasion track for epoxy-based and polyurethane-based coatings respectively. These micrographs reveal that epoxy-based and polyurethane-based systems show different behaviour.

On the one hand the epoxy nanocomposite coating EP A1 shows no significant changes in the surface morphology before and after 1000 cycles of the abrasion test. It seems that the larger SiC particles are located close to the coating surface, so that the wheels of the tester can only attach to the peak areas and the surrounding polymer matrix will be protected from direct contact with the wheels. This may explain the extremely low weight loss which has already been shown (*Table 1*).

On the other hand, in the case of the polyurethane-based coating system PU A9 there seems to be much more polymer matrix at the coating surface before the test. After the test, the SiC particles become more visible, an observation which indicates that a higher abrasion loss should have occurred. This result is in accordance with the higher weight loss for these systems (*Table 1*).

Thermal tests have little effect on contact angles

In addition to the abrasion resistance, which is an important basic property for materials used in industrial applications, their thermal and chemical stability is also significant. For dry processing in principle but also for wet processing, the coating layers should provide long-term stability with respect to their anti-adhesive properties even at elevated temperatures.

For this reason coated stainless steel plates were thermally treated in the dry state for 120 h at 120 °C and 150 °C respectively. The resulting contact angles are shown in *Table 2*. It appears that the dry temperature loading has almost no influence on the hydrophobic and oleophobic properties.

Chemical resistance may need to be enhanced

As regards chemical stability, it is important to note that appropriate cleaning procedures are usually obligatory in food processing equipment in order to meet hygienic requirements, even in cases where additional easy-to-

clean or anti-adhesive coatings have been applied on the surfaces that are in direct contact with the food.

For this reason it was of interest to investigate the stability of the coatings against attack by conventional cleaning agents with low and high pH values and at elevated temperature. The exposure to single cleaning agents should give a first indication of the suitability of the materials at the laboratory scale.

In more practical tests it is of course necessary that a combination of cleaning agents and some form of cycling should be tested to obtain reliable results for real applications. In addition, the food itself with its intrinsic chemical composition can have an influence on the stability of the coating layers and also has to be tested.

From Table 3 it can be concluded that the polyimides show the lowest chemical stability. In case of NaOH and HNO₃ at elevated temperature they are even destroyed by delamination. This behaviour is to be expected because the imide linking groups are sensitive to hydrolysis.

The epoxy systems also have some stability problems with HNO₃ attack. This can be explained by the fact that HNO₃ is an oxidising acid which probably destroys the amine linking groups in the epoxy network. The only matrix showing sufficient stability in these tests seems to be the polyurethane-based one.

Even the liquid products such as milk and carrot juice appear to have an effect on the contact angles. This is especially true for the contact angle against water (hydrophobic behaviour) which shows a more pronounced decrease than the contact angle against hexadecane (oleophobic behaviour) in the case of the nanocomposite coatings. The contact angles of the PTFE seem to be almost unaffected. However, it should be noted that the PTFE coatings showed significant defects after more than 48 h exposure and the tests were stopped at this stage.

Deposition and cleaning tested with vegetable juice

In order to obtain more preliminary information about the fouling and cleaning behaviour, laboratory experiments with thermal treatment of liquid products were performed under controlled conditions at IUV Bremen using a laboratory size heat exchanger.

For the experiments with carrot juice, the liquid testing substance was preheated to 70 °C and brought into contact with the coated and uncoated sample surfaces having a surface temperature of 115 °C with a flow rate of 6 cm/s. The fouling behaviour in this type of thermal treatment of carrot juice and the cleaning behaviour in laboratory experiments are shown in Figure 7.

The highest deposition could be observed for the PTFE-coated surface and stainless steel whereas the deposition for system PU A9 was comparable to electropolished stainless steel. For cleaning, the deposition layers of the deposits were swollen in 2 % NaOH and removed with a rubber spatula. The cleaning times decreased in the order stainless steel - electropolished stainless steel - polyurethane nanocomposite - PTFE as can be observed from Figure 7 (right). It is important to note that the coated surfaces perform much better in their cleaning behaviour than the stainless steel even in the electropolished case.

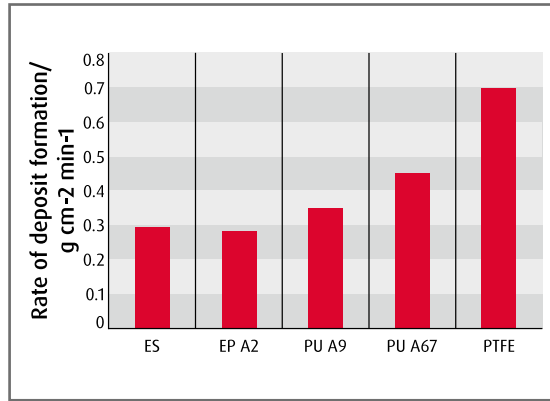


Figure 8: Deposition rates of whey proteins (10 % solution) on internally heated tubes in a flow channel, tube surface temperature 134 °C

Milk protein deposits are easily cleaned off

The same investigation was carried out for the fouling and cleaning behaviour using thermal treatment of whey proteins (Figures 8 and 9) as a reference for milk. Here another experimental set-up was chosen using a special flow channel in order to simulate the thermal process with higher flow rate and higher surface temperature, which would have more practical relevance.

Nanocomposite coated and uncoated stainless steel tubes that could be internally heated were placed in the flow channel. The surface temperature of the tubes

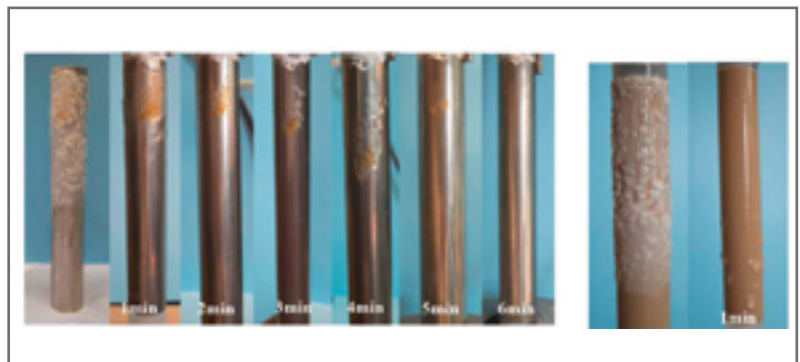


Figure 9: Cleaning behaviour of whey protein deposits in NaOH flow: stainless steel (left) and polyurethane nanocomposite coating PU A9 (right)



Figure 10: Heat exchanger plates coated with nanocomposite anti-adhesive systems after 4 h test in the heat exchanger at LUFA Norwest, left: epoxy nanocomposite EP A2, right: polyurethane nanocomposite PU A10

Table 3: Chemical stability tests: contact angles after maximum testing time before start of deterioration in relation to type of cleaning agent and food testing substance; PTFE-coated stainless steel; EP: epoxy matrix; PU: polyurethane matrix; PI: polyimide matrix; HD = hexadecane

System	Initial contact angle water/HD [°]	Contact angle after X h 2 % NaOH pH=13/90 °C [°]	Contact angle after X h 2 % HNO3 pH=1/90 °C [°]	Contact angle after X h milk 90 °C [°]	Contact angle after X h carrot juice 90 °C [°]
EP A1	101 / 65	24 h: 42 / 78	24 h: destroyed	48 h: 83 / 36	48 h: 58 / 55
EP A2	100 / 64	48 h: 58 / 70	24 h: destroyed	96 h: 72 / 37	96 h: 62 / 37
PU A9	103 / 64	48 h: 42 / 42	24 h: 96 / 56	96 h: 96 / 60	96 h: 96 / 60
PU A67	107 / 65	72 h: 100 / 62	96 h: 94 / 60	96 h: 88 / 57	96 h: 93 / 60
PI A5	106 / 66	24 h: destroyed	24 h: destroyed	24 h: 81 / 61	24 h: 73 / 31
PI A29	101 / 68	24 h: 80 / 57	24 h: destroyed	24 h: 84 / 57	24 h: 84 / 56
PTFE	118 / 55	48 h: 86 / 52	48 h: 77 / 51	48 h: 118 / 55	48 h: 115 / 54

was set to to 134°C and the flow rate was chosen to be 20 cm/s.

For the whey proteins, the deposition behaviour (Figure 8) is slightly different from the carrot juice. In this case the deposition rate is highest for the PTFE coated surface. The deposition rate for the epoxy system is quite comparable to stainless steel, while that for the polyurethane nanocomposites is slightly worse.

Cleaning behaviour with whey proteins was tested for the uncoated and coated stainless steel. For cleaning, the tubes with deposits were rinsed with 0.1N NaOH solution at ambient temperature using a flow rate of 0.6 m/s. The cleaning efficiency was checked every minute (Figure 9). It is clear from Figure 9 that the cleaning of the deposited whey proteins was much easier on the nanocomposite coated surface, which indicated the potential for improvement of the cleaning situation compared to stainless steel.

More demanding tests reveal some weaknesses

In order to obtain more information about practical relevance, the systems EP A2 and PU A10 were coated on real stainless steel heat exchanger plates. The coated plates were integrated into a heat exchanger system at LUF A Northwest technical area to test their performance in the thermal processing of milk with intermediate cyclic cleaning steps under real usage conditions.

A solution with 10 wt.% whey proteins and a volume stream of 1.5 l/min were chosen as standard test conditions. The temperature gradient was set at 85°C to 5°C. After 4 h the test was stopped and the plates were removed for visual inspection and gravimetric determination of deposits.

After this time, epoxy system A2 showed a deposit of 0.15 +/- 0.05 g and polyurethane system A10 only showed deposits of 0.08 +/- 0.03 g, whereas the deposits on stainless steel heat exchanger plates were determined to be 0.16 +/- 0.06 g, which in all cases was quite low. Additionally the coated heat exchanger plates showed damage at the contact point between adjacent plates caused by vibrations in the set-up. This damage is shown in Figure 10.

After six cycles each of 4 h processing and intermediate cleaning, some of the plates showed delamination of the coatings. Probably the combination of different influences such as high frequency vibration caused by the liquid flow inside the heat exchanger, the defects at the crossover points, the temperature gradient and the cyclic intermediate cleaning could have been responsible for the observed failure, a behaviour which had not been detected in the single-stress laboratory experiments earlier.

In further developments, the focus will be set on increasing the glass transition temperature of the matrix in order to minimise the creeping behaviour. This is probably responsible for the observed defects at the crossover points between the plates. Alternative pre-treatment methods for the stainless steel plates will also be examined in order to avoid sub-surface migration and delamination.

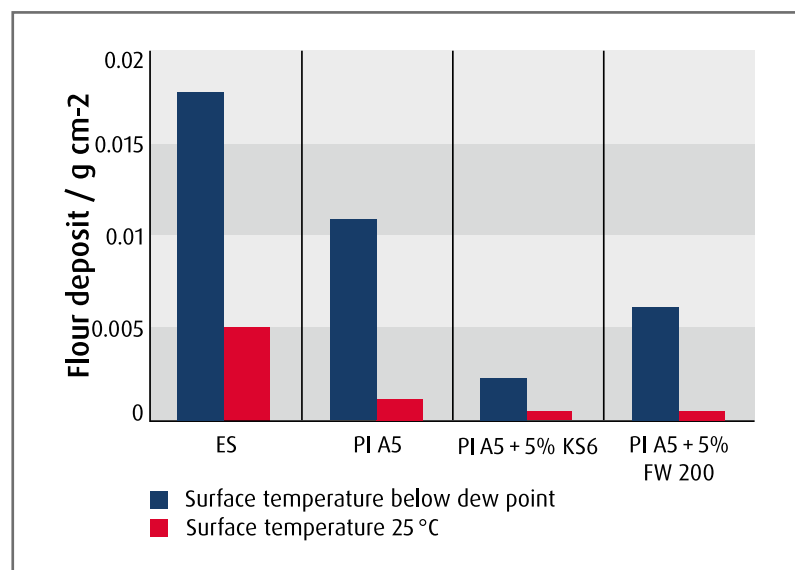


Figure 11: Gravimetric determination of flour (type 405) adhering to 1 m long stainless steel test tubes with 120 mm diameter at ambient temperature and below the dew point; ES: uncoated stainless steel, PI A5, PI A5 + carbon black FW 200, PI A5 + graphite KS6

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VINCENTZ

Table 4: Influence of modifier (FW 200 carbon black / KS 6 graphite) and modifier amount on static contact angles of polyimide coating against water and hexadecane (HD), surface roughness, and conductivity (determined by decay time and resistivity)

Type of modifier	Modifier amount, wt. %	Static contact angle [°]		Surface roughness [µm]			Decay time of electric-charge [s]		Resistivity [Ω]
		Water	HD	Ra	Rz	Rmax			
-	0	106	66	0.26 ± 0.07	1.63 ± 0.32	2.27 ± 0.85	2.9	5.1 x 10 ⁹	
	2.5	106	63	0.70 ± 0.10	4.21 ± 0.50	5.70 ± 0.82	0	1.3 x 10 ⁵	
	5	107	63	0.75 ± 0.03	4.76 ± 0.47	7.75 ± 1.92	0	1.2 x 10 ⁶	
	10	110	66	0.38 ± 0.096	2.85 ± 0.69	5.45 ± 3.09	0	1.5 x 10 ³	
	2.5	106	63	0.41 ± 0.10	2.64 ± 0.74	4.08 ± 1.49	0	2.3 x 10 ⁹	
	5	107	63	0.24 ± 0.06	1.53 ± 0.40	1.87 ± 0.66	0	1.1 x 10 ⁵	
KS 6	10	110	66	0.32 ± 0.01	2.06 ± 0.03	2.71 ± 0.29	0	8.7 x 10 ⁴	

Antistatic requirements for dry processing readily met

With respect to dry products, it was of particular interest to investigate the antistatic properties of polyimide systems suitable for dry product processing. In dry processes involving food production such as flour milling it is important to provide coating systems with antistatic behaviour in order to prevent explosions caused by electrostatic discharge phenomena.

Carbon fillers such as carbon black ("FW 200" grade) and graphite ("KS 6" grade) were dispersed in different amounts in a selected polyimide nanocomposite

system in order to elucidate the optimum concentration of these additives for an electrostatic effect while maintaining the abrasion stability of the coatings at the same time. Table 4 shows the results of the charge decay time and the resistivity in combination with the surface roughness for the two different additives in relation to the concentration.

From Table 4 it can be concluded that from a concentration of 5 wt.% the resistivity indicates antistatic behaviour of the coating systems. The charge decay time had already fallen to 0 s at a concentration of 2.5 wt.% carbon additive. The graphite filler in particular seems to have almost no adverse effect on the roughness of the coatings. The maintenance of a smooth surface is an important advantage in practical applications.

It seems that the required anti-static behaviour could be achieved using even small amounts of anti-static additive. The roughness could be kept to a low level, which is good basis for powder processing.

Surface adhesion of flour is greatly reduced

It was also of interest to perform more practical tests on small tubes in a pilot facility. In this type of experiments it is important to control the humidity in order to distinguish between different adhesion behaviours under dry and humid conditions. For this reason, measurements were performed at ambient temperature and at a temperature below the dew point.

The flour was transported together with a temperature controlled air stream in an aerocyclone set-up through internally coated tubes with 1 m length and 120 mm diameter. The results on the quantitative amount of adhering flour are shown in Figure 11.

As can be concluded from Figure 11, there is much less flour material adhering to the nanocomposite coated tubes than to the uncoated stainless steel tubes. The unmodified polyimide A5 shows significant improvement in dry and humid adhesion (below the dew point) compared to stainless steel.

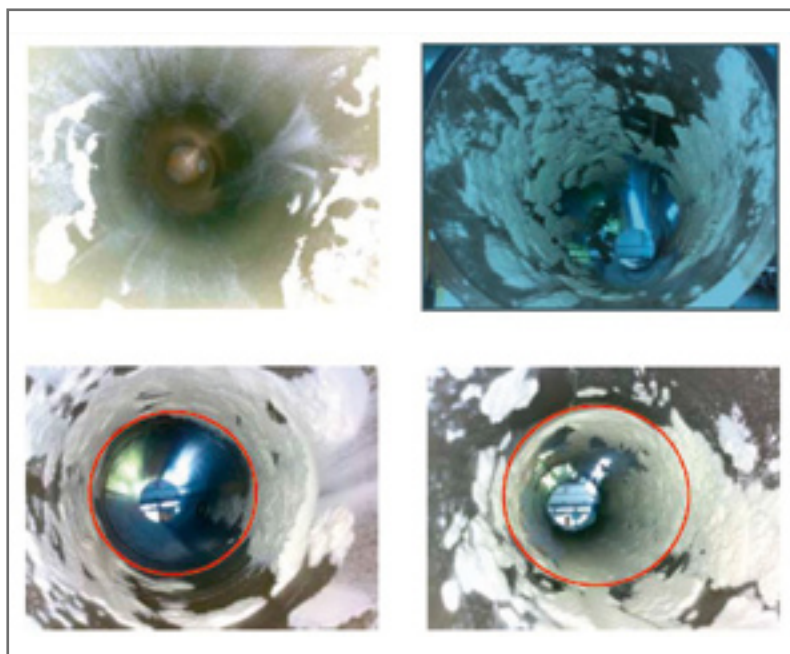


Figure 12: Inside view of testing tubes for gravimetric determination of flour adhesion; left side: PI A5 + graphite KS6; right: stainless steel, upper row: at ambient temperature, lower row: below the dew point, inside red ring = surface area analysed

In addition, the use of the carbon anti-static additives gives a more pronounced effect even though the surface roughness of the modified polyimide nanocomposites is slightly higher than that of the uncoated stainless steel, which could facilitate mechanical trapping.

Figure 12 shows photographs of the inside of the tubes directly after the test. In the case of experiments below the dew point, the tubes were temperature controlled in an area which is marked with a red circle. The end regions of the tubes were not controlled. For quantitative determination of the amount of deposited material the flour was collected only in the temperature controlled area and was quantified by weight (Figure 11).

It can be clearly seen that the deposits in Figure 12 correspond well to the values in Figure 11. From this point of view it seemed that very interesting anti-adhesive systems have been developed showing a great potential to improve the cleaning situation considerably.

A good basis for high performance coatings

These investigations revealed that interesting polymer-based anti-adhesive nanocomposites could be created which show favourable lower deposition levels than PTFE surfaces, with quite similar cleaning behaviour. The cleaning behaviour was also much better than that of stainless steel and electropolished stainless steel.

Their key advantage compared to PTFE is their ability to be applied by spray coating on almost every type of substrate and to be cured under comparatively mild conditions. Although the chemical stability in a combined wet and hydrolytic environment is still a challenge for these systems, they show excellent behaviour in relation to applications in dry processes, as could be shown for the polyimide-based coatings.

For wet processes, there is still potential for optimisation, and ongoing experiments seem to be very promising in this direction. It is important to note that antistatic properties can be added without losing mechanical stability. Overall a materials basis has been worked out which after appropriate optimisation and technology development has major potential for use as an anti-adhesive surface in industrial processes. ◀

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Results at a glance

» A new class of anti-adhesive coating materials can be used to change the wetting behaviour of internal surfaces of machinery used in the food production industry. They can be spray-applied and cured at lower temperatures than the PTFE coatings now widely used.

» The systems are based on a polymer binder matrix filled with inorganic nanoscale SiC particles to provide abrasion stability and containing perfluorinated cross-linkable co-monomers to decrease the surface free energy.

» Both epoxy and polyurethane matrix-based coatings gave high abrasion resistance and good alkali resistance. Only the polyurethane system gave reasonable resistance to other materials (carrot juice and milk at 90 °C, nitric acid).

» The polyimide-based systems performed well in dry food processing tests. The necessary antistatic properties could be controlled by use of carbon or graphite additives.