



DURABLE WATER-BORNE WOOD COATINGS

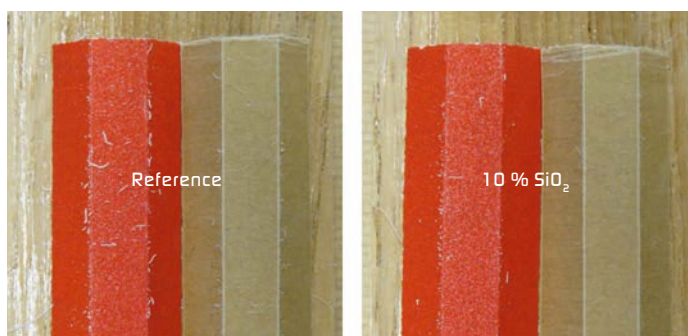
Adding silane-modified colloidal silica enhances performance of water-borne clear coatings. By Peter Greenwood, Levasil Colloidal Silica - AkzoNobel Specialty Chemicals.

When making the switch from solvent-borne to water-borne coatings the goal is to not compromise the function and performance of wood coatings. The effect of using epoxy silane-modified colloidal silica in water-based clear coatings on open time and anti-blocking, sanding and mechanical properties was investigated.

Many researchers have investigated the use of silica nano-particles in resin-based systems. Water-free systems of silane-modified colloidal silica particles have been found to enhance mechanical properties of coatings [1, 2]. Silane-modified fumed silica has been used to enhance coating properties [3]. Organosols have been used to improve scratch resistance in clear coatings by surface enrichment [4]. Aqueous colloidal silica has been used in co-polymerisation of resins [5]. Co-polymerised colloidal silica resin hybrids are also used to enhance a variety of coating properties like hardness, anti-blocking and dirt-pickup [6]. Silica particles made from tetraethyl orthosilicate (TEOS) have recently been investigated in co-polymerisation of hybrid coatings, where hardness and adhesion properties were found

to be improved [7]. Non-surface-modified colloidal silica has been tested as a nano-filler in latex coatings with encouraging results for mechanical properties [8].

Figure 1: Sandability - sandpaper P180 (red) and P240 (brown).



RESULTS AT A GLANCE

- The effect of adding epoxy silane-modified colloidal silica particles to water-based clear coatings depended greatly on the type of resin and other components in the formulation.
- Improved hardness and significantly improved block resistance (and sanding properties) were observed.
- Results for extending the open time were positive but heavily dependent on formulation and type of rheology modifier.
- Silane modification had good compatibility with resins and did not compromise aesthetic coating properties (gloss and haze).
- Adding colloidal silica to water-borne wood lacquers enhanced the grain structure and appearance in a manner similar to solvent-borne lacquers.

Figure 2: Grain accentuation – Enhancement of grain structure by wood lacquers. 1-K water-based lacquer formulation No 7 with (right) and without (left) 20 % SiO_2 addition of epoxy-silane modified colloidal silica.



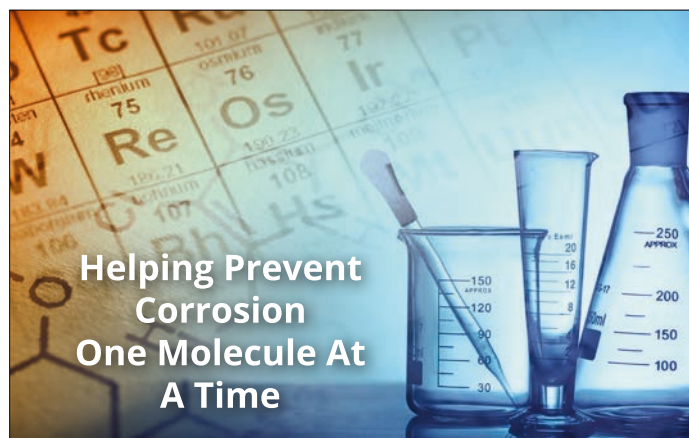
of added silica). The best results to date have been obtained with urethane thickeners (Newtonian flow type) combined with silane-modified colloidal silica. It would be of great interest to evaluate and verify whether silane-modified colloidal silica could work in a similar way for clear transparent coatings.

IMPROVING MECHANICAL PROPERTIES

Very few studies have been done on the use of silane-modified water-based colloidal silica particles in formulations of water-borne coatings. Recently there has been great interest in such particles in water-borne lacquers since they have shown to provide benefits like anti-blocking, sanding and wood grain accentuation in acrylic emulsion-based wood coatings. Highly cross-linked systems like two-pack (2-k) systems are often the best solution for improving mechanical properties. One of most readily available sources of nanoparticles for coatings is concentrated silane-modified colloidal silica in the form of aqueous sols. Compared with conventional silica sols, silane-modified colloidal silica has greater stability towards aggregation and gelling, both as is and in latex-based coating formulations [9, 10, 11].

LOWERING VOC

Resins contain VOC, so from an environmental point of view, using colloidal silica to replace part of the resin in latex-based coating formulations is advantageous. Softer resins with less need for coalescing agent can be used since colloidal silica addition will improve mechanical properties and remove the need to use potentially hazardous film-forming agents such as NMP or glycol ethers. Additionally, extending open time has been of increasing importance since VOC legislation makes it useful to reduce film-forming agents. This property is very important for highly concentrated pigmented systems such as paints but also for clear/transparent coatings for e.g. flooring applications such as parquet lacquers. It has been found that surface-modified colloidal silica is efficient as an open-time extender for pigmented water-based paints [12]. Its efficiency depends on paint composition (the type of thickening agent and the amount



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Table 1: Gloss and Haze.

Formulation	Gloss 20°			Haze index (Gloss 60° – Gloss 20°)		
	Reference	10% SiO ₂	20% SiO ₂	Reference	10% SiO ₂	20% SiO ₂
No 8	127	130	112	57	56	8
No 9	108	99	129	60	57	50
No 10 (1-k) (reference)	15			21		
No 10 (2-k) (reference)	18			26		
No 11 (1-k)		19*			27*	
No 11 (2-k)		21*			27*	

*: 14% SiO₂

Table 2: Block resistance results for clear 1-pack acrylic formulations.

Formulation	Block resistance	Example formulations		
		Raw material	No 1 reference	No 2 10% SiO ₂
No 1 - reference	0	Setaqua 6776	87.1	87.1
No 2 - 10% SiO ₂	4	Dehydran 1293	0.3	0.3
No 3 - reference	2	Rheolate 212 solution	0.6	0.6
No 4 - 10% SiO ₂	4	Dowanol DPnB	5.7	5.7
No 5 - reference	3	Water	5.6	0
No 6 - 10% SiO ₂	5	Levasil CC301	0	12.47

Ranking: 0 = worse, 5 = best

Table 3: Taber Abrasion resistance – weight loss mg.

Formulation	Number of revolutions			Number of revolutions			Number of revolutions		
	500			1000			1500		
No 10 (1-k) reference	5			12			17		
No 10 (2-k) reference	8			16			25		
No 11 (1-k) 14% SiO ₂ addition	8			21			34		
No 11 (2-k) 14% SiO ₂ addition	5			10			21		
Formulation	Reference			10 % SiO ₂ addition			20 % SiO ₂ addition		
	Number of revolutions			Number of revolutions			Number of revolutions		
	100	500	1000	100	500	1000	100	500	1000
No 8	3	33	60	2	17	37	3	33	64

RESULTS

The effect of adding silane-modified colloidal silica to water-based clear coating formulations on open time and on the anti-blocking, sanding and mechanical properties of resin films was studied. Twelve different varnish formulations were used on PUD resin in the testing: (a) 1- and 2-pack acrylic formulations basically containing resin, modified-colloidal silica, coalescing agents and additives and (b) fully formulated clear floor coatings. Before testing, all the samples were conditioned at 23°C and 50 % RH for 24 hours.

AESTHETIC PROPERTIES NOT COMPROMISED

Table 1 shows little or no change in gloss when silica is added (even some increased gloss), indicating good silica compatibility with the resin. Gloss (20°) was measured on coatings applied to aluminum plates after 30 days of drying at ambient temperature at dry film thicknesses between 40 and 50 µm. Haze index, defined as a gloss difference, was calculated from gloss measurements at 20° and 60° following ASTM D4039 (Reflection haze of high-gloss surfaces). The gloss of the varnishes was determined as the average of five measurements.

SIGNIFICANT ENHANCEMENT OF BLOCK RESISTANCE

Adding silane-modified colloidal silica to aqueous systems was beneficial for blocking resistance. The best results were obtained for the system with highest tack (Formulation 1 and 2 in Table 2). Block resistance was evaluated according to ASTM D4946 'Standard test method for Block Resistance of Architectural Paints'. The wood coated parts were placed face to face with a load weight of 1 kg (127 g/cm²) at 60 °C for 30 minutes after 24 hours of drying at room temperature.

BENEFICIAL FOR SANDING PROPERTIES

Adding silane-modified colloidal silica was beneficial for sanding properties. Figure 1 shows that the damage done by sand paper is less on a coating including colloidal silica than for the coating without colloidal silica. A likely explanation is that a less tacky coating does not plug and damage the sanding paper to the same extent. The sandability of the different coatings was estimated according

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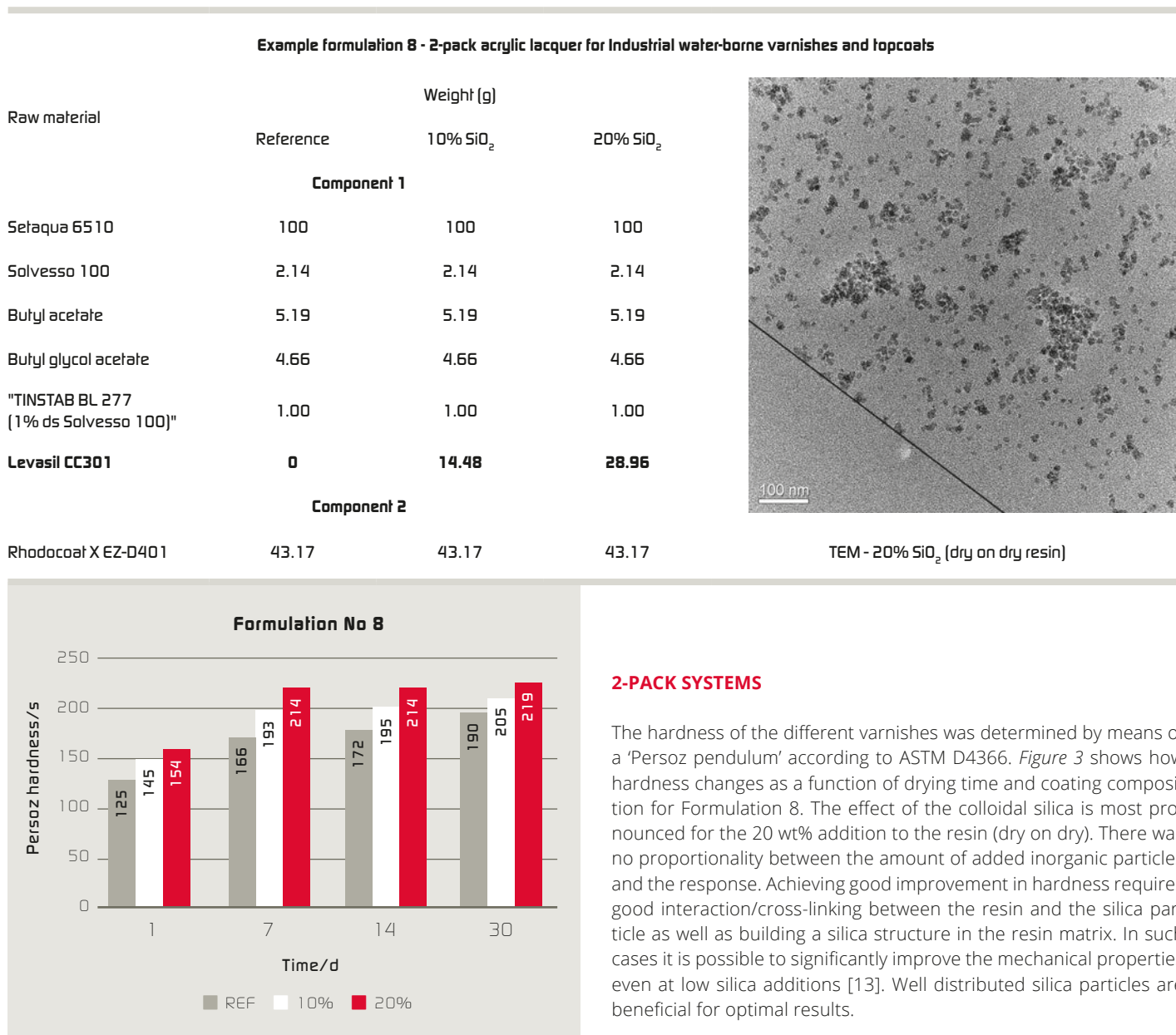
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Figure 3: Persoz hardness as a function of coating composition and drying time at ambient temperature for formulation No 8.



- to the ISO 11998 standard 'Determination of wet-scrub resistance and cleanability of coatings'.

KEEPING THE SOLVENT-BORNE LOOK

It has been reported in literature that water-based wood coatings comprising colloidal silica, organosilane and latex emulsion can penetrate into the wood substrate [13]. Adding epoxysilane-modified, 7 nm silica particles, to water-borne 1-pack coating formulations containing acrylic binders warmly enhanced the wood grain structure (*Figure 2*) giving an appearance similar to solvent-based coating systems. This valuable aesthetic effect can also be expected to be present with water-borne 2-pack coating systems based on acrylic binders to which silica particles have been added, combined with the excellent mechanical properties characteristic of such systems. PUD based systems will show similar benefits from silica addition for both 1-pack and 2-pack coatings.

SIGNIFICANT IMPROVEMENTS IN MECHANICAL PROPERTIES IN

2-PACK SYSTEMS

The hardness of the different varnishes was determined by means of a 'Persoz pendulum' according to ASTM D4366. *Figure 3* shows how hardness changes as a function of drying time and coating composition for Formulation 8. The effect of the colloidal silica is most pronounced for the 20 wt% addition to the resin (dry on dry). There was no proportionality between the amount of added inorganic particles and the response. Achieving good improvement in hardness requires good interaction/cross-linking between the resin and the silica particle as well as building a silica structure in the resin matrix. In such cases it is possible to significantly improve the mechanical properties even at low silica additions [13]. Well distributed silica particles are beneficial for optimal results.

WELL DISTRIBUTED SILICA PARTICLES ARE BENEFICIAL

Transmission electron microscopy (TEM) was used to visualize the structure of the inorganic part embedded in the organic matrix. *Figure 3* shows a TEM picture showing the inorganic structure embedded in Setaqua 6510 (Formulation No 8) varnish with a 20 % colloidal silica content. The resin – air interface is marked by a diagonal line in the bottom left of the image.

THE EFFECT ON ABRASION RESISTANCE IS FORMULATION DEPENDENT

The resistance to abrasion of the different varnishes with and without addition of epoxy silane-modified colloidal silica applied to aluminum plates, at dry film thicknesses between 40 and 50 µm, was determined by means of a Taber abraser following ASTM D4060. The effect of silica on abrasion resistance is very formulation dependent. The silica distribution in the matrix and the degree of cross-linking in the matrix are important factors; i.e. there is a big difference between one-pack and two-pack systems. Abrasion resistance was reduced in one pack-systems, likely due to increased friction coef-

ficient (though not measured in this specific case) while it was significantly improved in two pack-systems, compared with Formulations 10 (reference) and 11 in *Table 3*. Interestingly, using 'Bayhydur/Proglyde' solution in the 2-k formulation appears to make the reference system less resistant to abrasion compared to the reference system based on a PU dispersion only. Once again, one explanation could relate to the system's increased friction coefficient/tackiness. For Formulation No 8, there is improvement at 10 % silica but some deterioration with 20 %.

EXTENDED OPEN TIME

Extended open time was system- and concentration-dependent. Open time (the coalescence process) can be affected by several factors, e.g. the Tg of the polymer, the coalescing agent content (MFFT), the nature and type of the stabilization of the polymeric particles (anionic, non-ionic), polymer particle size and particle size distribution, additives such as waxes, dispersing agents, anti-foaming/defoaming agents and thickeners (nature and added amount). All those factors impact the coalescence process and then affect the open time to varying degrees.

In *Table 4* the clear-coat systems, formulations No 12 based on Alberdingk AC2736 resin, clearly show increased open time in presence of silane-modified colloidal silica. The improvement depends strongly on the colloidal silica content. The brush method appears to indicate a longer open time and a greater increase with silica addition both in absolute values and as a percentage relative to data obtained with the drying time recorder. Open times were determined according to ASTM D7488 'Standard test method for open time of latex paints'.

In parallel with determinations by means of the brush test method, open times have also been evaluated by a method using a drying time recorder according to ASTM D5895. For Formulation 12, open time can be doubled by adding 10 % Levasil CC301. This system also had higher values for the brush test compared to those obtained by the drying time recorder method. Good correlation was obtained between the two test methods. One likely explanation for the extended open time could be reduction in skin formation, since colloidal silica by itself is not a film-forming material at 'normal' temperatures.

INFLUENCE ON OPEN TIME AT CONSTANT VISCOSITY

To evaluate the impact on open time at constant viscosity, viscosities were adjusted by increasing the amount of thickener for two types of thickeners, HEUR and EHEC. Combinations with HEUR thickeners appeared to be beneficial for extending open time, which was not the case with EHEC thickeners. This is in line what has been reported for paints [12].

INFLUENCE ON OPEN TIME FOR PARQUET LACQUER FORMULATIONS

For the parquet lacquer formulations based on PUD resin, similar results were seen for open time as for the clear wood stain formulations (*Table 5*). However, in this case open times determined by the drying recorder method tended to be higher compared to those obtained according to ASTM D7488 (brush test method). Use of colloidal silica significantly extended the open time; it did not seem to affect surface tension. Dynamic surface tensions were determined for all formulations to be about 28 mN/m throughout the drying process. So increased open time was not re-



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- lated to changing surface tension during the drying process. The viscosity profiles were similar for the parquet lacquer formulations. This also supports the hypothesis that the increased open time is related to reduced skin formation caused by the presence of silica at the coating surface.

CONCLUSIONS

The effect of the silica particles depended strongly on the resin and other formulation components. Significantly improved block resistance was observed, hence sanding properties, as was improved hardness. The effect on abrasion resistance was less clear but still noticeable in many coating systems. The changes were compatible with the resins and aesthetic properties (gloss and haze) were not compromised; for some formulations they even improved. Open time was increased significantly in clear coating formulations but results were heavily dependent on formulation and type of rheology modifier. 

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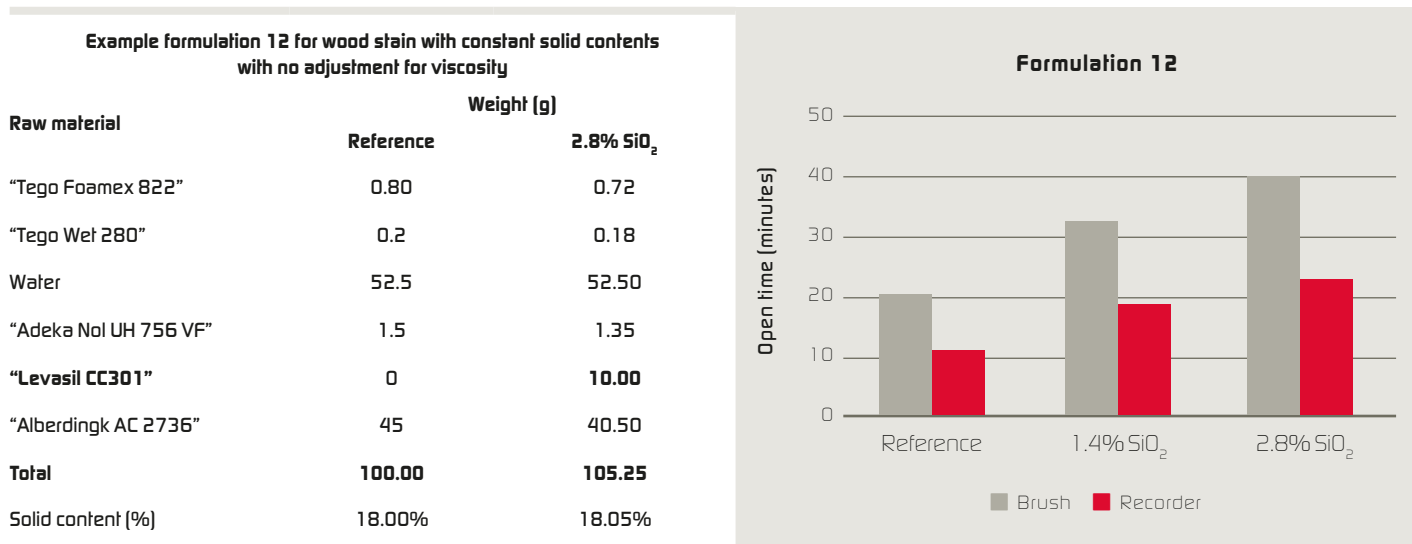
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Table 5: Open time for PUD parquet lacquer formulations.

Formulation	ASTM D7488 (minutes)	ASTM D5895 (minutes)
No 10 (1-k) reference	14-16	21
No 11 (1-k) 14% SiO ₂	20-22	25
No 10 (2-k) reference	12-14	14
No 11 (2-k) 14% SiO ₂	16-18	19

Table 4: Open time - Influence of colloidal silica on open time at constant solid contents. Open time by brush method (ASTM D7488) and drying time recorder (ASTM D5895).



“The main function from colloidal silica is reduction in tackiness of the paint film.”

3 questions for Peter Greenwood

Can silane-modified colloidal silica improve further properties of water-borne coatings?

Silane modified colloidal silica is also used in water-borne exterior paints and coatings for enhancement of dirt-pick-up resistance. The main function from colloidal silica is reduction in tackiness of the paint film and thereby you can achieve better resistance towards both hydrophobic and hydrophilic dirt.

Would the observed improvements also apply to other coatings systems (than wood)?

Improvement in mechanical performance is related to the crosslinking between silica and resin in two-pack systems and is a general feature that can be applied to many types of surfaces. Enhancement of block resistance is another general feature than can be used in e.g. roof tile coatings where stackability is crucial.

Will you investigate the addition of silane-modified colloidal silica in combination with further resin systems? *At the moment we are not doing any further investigations on wood coatings since we are focusing our development on elastomeric cool roof coatings and are planning to submit a paper on the topic for ECS 2019.*



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