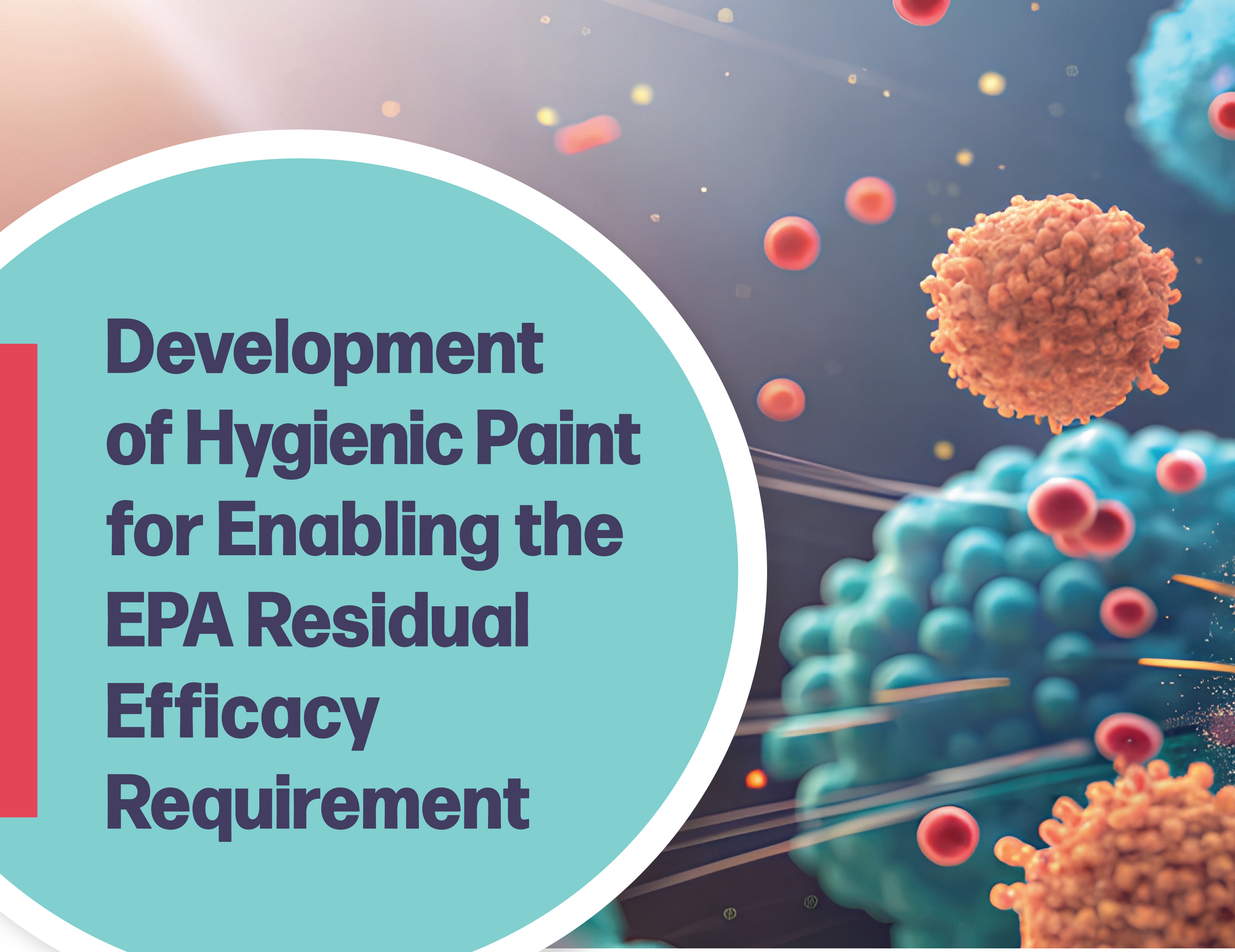

Development of Hygienic Paint for Enabling the EPA Residual Efficacy Requirement

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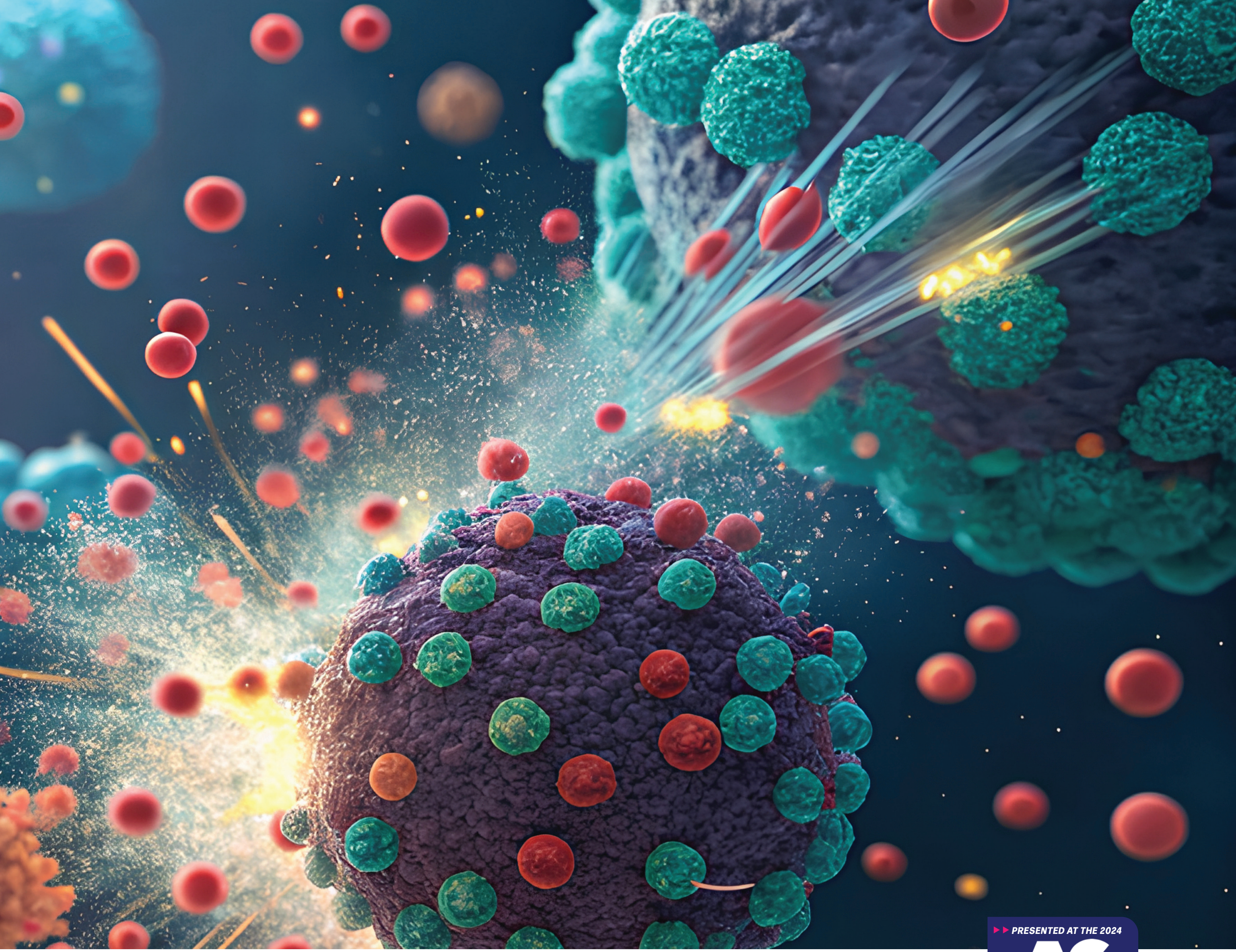
Over the past couple years, hygienic paints and coatings have gained significant attention due to their continuous protection against the COVID-19 virus. Recently, the EPA published its interim guidance on the "Review for Products Adding Residual Efficacy Claims," which requires ≥ 3 Log10 reduction of bacteria and virus after chemical treatment and dry abrasion in order to claim residual efficacy of paints and coatings. In this article, the development of hygienic paints using a proprietary insoluble copper (iCopper) and hydrophilic-modified DDAP/DDAB quaternary ammonium compound (Quat) technologies is presented. The antibacterial and residual efficacies per the Japanese Industrial Standard JIS Z2801 and the EPA interim method have demonstrated that the iCopper technology can achieve residual efficacy of ≥ 4 Log10 reduction in representative latex paints. In addition, the data have shown the full compatibility of the iCopper technology with latex paints and its impact on paint color can be further minimized by formulation optimization. Other dry film properties such as scrub and stain resistance were also tested and results did not indicate any negative impact from the iCopper technology. Conversely, DDAP/DDAB Quats give < 1 Log10 reduction of bacteria in latex paint per the JIS Z2801 test method even though Quat is an important biocidal active in disinfectant products. This finding may be attributed to the limited mobility of Quat molecules in the dry film as a result of strong electrostatic attraction between cationic Quat and anionic latex particles. The data suggests a significant paint reformulation is required to enable good efficacy of DDAP/DDAB Quat. In summary, the iCopper technology would provide a more cost-effective viable solution to meet the EPA residual efficacy claim for hygienic paints and coatings.

Introduction

Microbes such as the novel SARS-CoV-2 virus can survive on surfaces, which facilitates their acquisition and transfer to humans. As a result, hygienic paints and coatings have gained significant attention in the past couple years for their continuous protection against the COVID-19 virus. In early 2021, EPA published their interim guidance on the "Review for Products Adding Residual Efficacy



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Claims."[1] Hygienic paints and coatings fall into the category of "Supplementary Residual Antimicrobial Products." For a residual efficacy claim to be supported, a $\geq 3\ \text{Log}_{10}$ reduction of bacteria (*Staphylococcus aureus, Pseudomonas aeruginosa*) and viruses (most difficult to kill) after chemical and dry abrasion must be achieved.

Copper/silver and quaternary ammonium compounds (Quats) are well-known for their antibacterial and antiviral efficacies.[2,3] Currently, there are only a few hygienic paints on the market. For example, PPG recently developed an architectural paint using a cuprous/Cu(I)-based technology from Corning,[4] while Sherwin-Williams launched an antimicrobial paint derived from alkyl dimethyl benzyl ammonium chloride (ADBAC) Quat.[5] However, significant challenges still exist for developing copper or Quat-based hygienic paints that can meet the EPA residual efficacy claim. Copper technology must have good compatibility with acrylic paints while its impact on color needs to be mitigated. Conversely, cationic Quats are normally incompatible with latex paints, the latter of which are stabilized by anionic surfactants. Therefore, paint reformulation is sometimes required to accommodate Quat(s), which is time- and effort-consuming.[6]

In this article, we report on the development of a hygienic paint using patent-pending insoluble copper (iCopper)[7] and proprietary hydrophilic-modified DDAP/DDAB Quat technologies to meet the EPA residual efficacy claim. Antibacterial efficacies were evaluated by both the Japanese Industrial Standard JIS Z2801 and the EPA interim method to assess their initial and residual efficacies. Potential mechanisms of actions will be proposed to explain the observed efficacies. Other dry film properties, such as color, scrub, and stain resistance, were also tested to ensure no negative impacts resulted from the iCopper technologies.

In this article, we report on the development of a hygienic paint using patent-pending insoluble copper (iCopper) and proprietary hydrophilic-modified DDAP/DDAB Quat technologies to meet the EPA residual efficacy claim.

Experimental Procedure

Preparation of Hygienic Paints

The iCopper and DDAP/DDAB Quat technologies were evaluated in one prototypical interior flat paint (50% PVC, 35% Solid Vol, 45 g/L VOC, **Table 1**) prepared internally and two commercial paints (one matte wall paint: 41.5% Solid Vol, < 50 g/L VOC; one semi-gloss interior paint: 37.45% Solid Vol, < 50 g/L VOC) were purchased and used as received. The iCopper product was post-added into the above paints followed by speed-mixing for homogeneity. The Cu level ranged from 165 to 1670 ppm. For comparison, 5% copper sulfate ($CuSO_4$) aqueous solution was also post-added into the paints to achieve 330 to 1165 ppm Cu level. For the Quat-treated paints, 5% aqueous solutions of DDAP/DDAB Quats and an ADBAC (ADBAC: alkyl (C_{14} 50%, C_{16} 10%, C_{12} 40%) dimethyl benzyl ammonium chloride) Quat benchmark were individually post-added into the paints to achieve 2000 ppm Quat level. All the treated paints were equilibrated overnight prior to any tests.

TABLE 1
Prototypical Flat Wall Paint Composition

Material	Weight (g)
Water	350.5
Defoamer	4.6
Dispersant	10.6
Surfactant	1.1
Ethylene glycol	29.3
Texanol	3.8
Cellulose	3.0
Attapulgite	5.9
pH adjuster	As Needed
Kaolin	87.8
Nepheline syenite	146.3
Titanium dioxide	152.1
100% Acrylic emulsion	205.0
Total Weight (g)	**1000.0**

Antibacterial Residual Efficacy Tests

The JIS Z2801 method was modified for evaluating the initial antibacterial efficacies of copper and Quat-treated paints by reducing the contact time from 24 to 2 h and replacing *Escherichia coli* (ATCC 8739) with *Pseudomonas aeruginosa* (ATCC 15442).[8] In brief, the paints were brushed onto sterilized plastic filter paper. Each side of the paper was painted twice, and the coated paper was dried at ambient conditions for 3 days. The dried coatings were then leached against 10 L water for 24 h (four water changes in first 8 h). Each of the two bacterial inoculum (0.4 mL) was used to inoculate the dry film (40 mm × 40 mm) of test paint to achieve 1.0 to 4.0 × 10^5 cfu on the dry coating surface. The test samples were covered by a film and incubated at 35 ± 1 °C and 90% relative humidity (RH) for 2 or 24 h. The degree of antibacterial efficacy was determined relative to blank paint via viable cell count of bacteria by the agar plate culture method.

The EPA interim method was applied for evaluating residual antibacterial efficacy.[9] In brief, the paints were drawn down onto a sterilized steel substrate (1 in. × 1 in. of AISI type 304 stainless steel). The coated substrate was dried at ambient conditions for at least 24 h. Some of the dried coated substrates were further subjected to dry and chemical abrasion for the 1-week residual efficacy claim. For dry abrasion, the coated substrate was exposed to 10 cycles of dry abrasion, each of which comprised 16 single passes by a Scotch Brite Non-Scratch Scrub Sponge using a BYK Gardner-Scrub machine. For chemical abrasion, the scrub sponge was soaked in sodium hypochlorite (2000 ppm NaOCl) solution and Nugen™ MB5N-256 (256X dilution) disinfectant prior to abrasion. The coated substrate was exposed to 10 cycles of chemical abrasion by the soaked scrub sponge, each of which comprised 8 single passes. After rinsing and drying to remove the residual NaOCl and Quat, each of two bacterial inoculums (20 uL), *Staphylococcus aureus* (ATCC 6538) and *Pseudomonas aeruginosa* (ATCC 15442), was used to inoculate the dry film of the test paint to achieve 10^4 to 10^5 cfu on the dry coating surface. The test paints were incubated at 22 °C and 30-40% RH for 2 h. The degree of antibacterial efficacy was determined via viable cell count relative to the uncoated substrate by the agar plate culture method.

Dry Film Property Measurement

Compatibility and Color

A drawndown film of 5 mil wet thickness was applied onto a sealed Leneta chart, and then dried at ambient conditions for 1 day. The resulting dry film was visually inspected for any defects due to potential incompatibility between Cu/Quat and latex paint and rated as follows:

- Rating of 2: Pass without any defects such as pinholes, fisheyes, or grits, etc.
- Rating of 1: Fail with some defects such as pinholes, fisheyes, or grits, etc.
- Rating of 0: Fail with many defects such as pinholes, fisheyes, or grits, etc.

The color (CIE L × a × b values) of the coating samples was measured by a BYK spectro-guide colorimeter and then compared with the reference. The color difference was calculated by $\Delta E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{0.5}$ (overall) and $\Delta E' = (\Delta a^2 + \Delta b^2)^{0.5}$ (color only). The color difference is considered not visually perceivable if $\Delta E/\Delta E' < 2$.

ASTM D2486 Scrub Resistance[10]

First, 6 mil wet thickness films of the treated and reference paints were simultaneously drawn down side-by-side perpendicular to the length of a plastic Byko chart. The films of the testing and reference paints were duplicated by reversing their positions on the panel. The coatings were dried at ambient conditions for 7 days prior to scrub tests. A nylon bristle brush was conditioned in water for 24 h prior to use. The brush was then removed and shaken vigorously to remove any excessive water. Ten grams of the stirred abrasive scrub medium from the Leneta Company was evenly added on top of the brush and 5 mL water was placed on the surface of the coatings along the path of the brush. The coating films were scrubbed at the rate of 37 cycle/min. The test was stopped after each 400 cycles before failure. The brush was removed for reapplying 10 g of the scrub medium, and 5 mL water was placed on the coating surface along the path of the brush. The testing was resumed until the failure of the coatings (one continuous thin line of 12.7 mm width of the shim). The numbers of cycles to failure were recorded for both reference and test coatings, and the percentage of scrub resistance retained for treated paint was calculated as follows:

$$\%\ Scrub\ Resistance = \frac{\#\ of\ Cycles\ to\ Failure\ of\ Testing\ Paint}{\#\ of\ Cycles\ to\ Failure\ of\ Reference\ Paint} \times 100\%$$

ASTM D4828 Stain Resistance[11]

Next, 3 mil wet thickness films of the testing and reference paints were simultaneously drawn down side-by-side, parallel to the length of a plastic Byko chart. The films of the test and reference paints were duplicated by reversing their positions on the panel. The coatings were dried at ambient conditions for 7 days prior to stain tests. Different stains were then applied perpendicular to the length of the paint drawdown. The panel was kept horizontally, and the stains were allowed to remain in contact with the coating for 1 h. Subsequently, the panel was placed on the washability machine and 1 mL Fantastik detergent was applied in parallel to each type of stain line. A sponge was soaked in water and squeezed with one hand until no more water dripped from it. The sponge was then placed in the holder and 15 mL water plus 5 mL Fantastik detergent were applied to the sponge. The sponge assembly was placed on one end of the washability machine and run for 100 cycles at the rate of 37 cycle/min. If all the stains were removed prior to 100 cycles, the test was stopped and the number of cycles was recorded. The testing panel was removed, rinsed under running water, and allowed to dry. The relative stain resistance of the test coating was determined by comparing with that of the reference on the same panel and rated as follows:

- (=): Equivalent stain resistance to the reference
- (sl+/sl-): Slightly better/worse stain resistance than the reference
- (+/-): Moderately better/worse stain resistance than the reference
- (++/--): Significantly better/worse stain resistance than the reference

Results and Discussion

The iCopper product and DDAP/DDAB Quats were leveraged for developing hygienic paints with residual efficacy. First, the iCopper and Quat technologies were inspected to ensure good compatibility in latex paints. Then, the JIS Z2801 antimicrobial test was performed to ensure good initial antibacterial efficacy prior to proceeding to the more challenging EPA interim method for residual efficacy. Finally, other dry film properties were tested to ensure no or minimal negative impacts.

Compatibilities of Copper/Quat-treated Paints

The compatibilities of the iCopper technology and soluble $CuSO_4$ in the prototypical paint were evaluated via visual assessment of dry film appearance. As shown in **Figure 1,** the iCopper product is compatible with the prototypical latex paint while $CuSO_4$ is not. U.S. Patent 4,517,330 demonstrated that soluble copper such as copper bisdimethylamino acetate can be used to crosslink acid-functional emulsion copolymer for enhanced durability.[12] Thus, the above observed compatibility can be attributed to water insolubility of the iCopper product that prevents the formation of copper carboxylate and thus the crosslinking of individual latex particles together.

Quats are known to be incompatible in latex paints due to the strong electrostatic attraction between cationic Quat and anionic acrylic/surfactants that results in an undesirable viscosity increase in wet paint and grit formation in dry films. To address this, we designed hydrophilic-modified DDAP/DDAB Quats that are significantly more compatible than, e.g., ADBAC, in the prototypical paint (**Figure 2**).

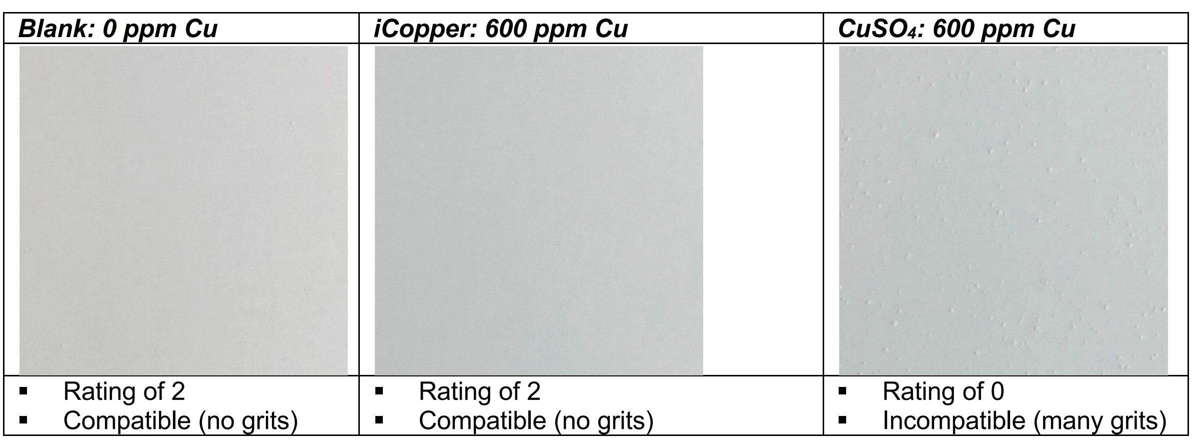


FIGURE 1 Compatibilities of copper compounds in latex paint.

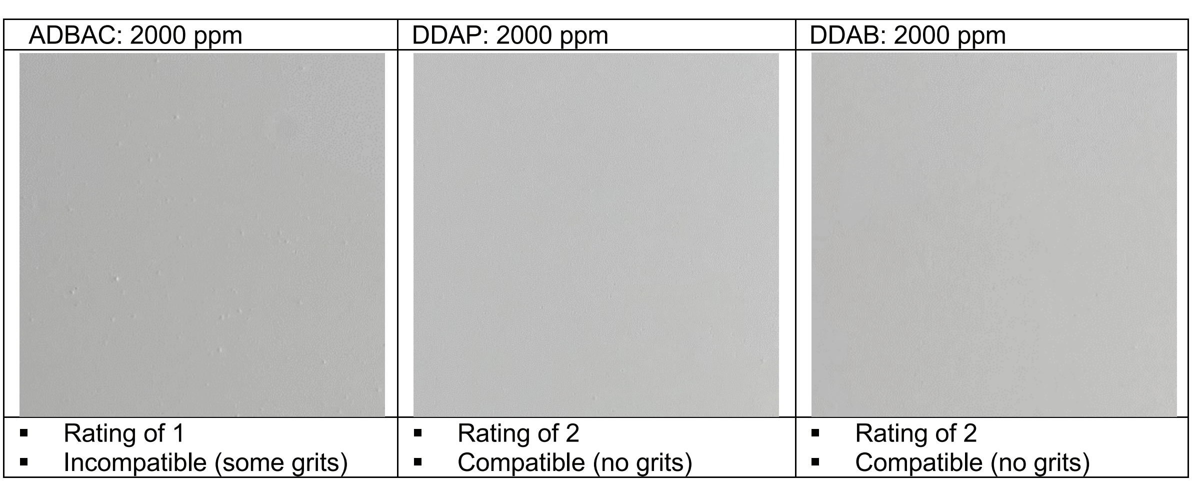


FIGURE 2 Compatibilities of Quats in latex paint.

Initial Antibacterial Efficacy per the JIS Z2801 Method

In light of the above compatibility results, only iCopper and DDAP/DDAB Quats were evaluated by the JIS Z2801 method for initial antibacterial efficacy in the prototypical latex paint. As shown in **Table 2,** the iCopper technology gives > 99.9% or 3Log10 reduction in bacterial counts at 824 ppm Cu and 2 h of contact time.

The antimicrobial efficacy of copper-based biocides is well known and can be attributed to multiple mechanisms, as summarized below.[4,13]

- Direct cell membrane damage
- Generation of reactive hydroxyl radicals through Fenton-type reactions resulting in the oxidation of proteins and lipids

$$Cu^{2+} + O_2^{\circ-} \rightarrow Cu^{+} + O_2$$

$$\text{Fenton } Cu^{+} + H_2O_2 \rightarrow Cu^{2+} + {}^{\circ}OH + OH^{-}$$

$$\text{Haber Weiss } O_2^{\circ-} + H_2O_2 \rightarrow O_2 + {}^{\circ}OH + OH^{-}$$

- Depletion of -SH in cysteines or glutathione in a cycle per the following reactions

$$2\ Cu^{2+} + 2\ RSH \rightarrow 2\ Cu^{+} + RSSR + 2\ H^{+}$$

$$2\ Cu^{+} + 2\ H^{+} + O_2 \rightarrow 2\ Cu^{2+} + H_2O_2$$

- Entry of Cu^{+}/Cu^{2+} ions into cells via ligand interactions causing disruption of RNA and DNA function

On the other hand, the DDAP/DDAB Quats give < 80% or 1 Log10 reduction in bacterial counts at as high as 2000 ppm level after as long as 24 h of contact time (**Table 3**). This is significantly less efficacious than the iCopper technology, even though Quats are widely used in hygiene as effective disinfectants. Since the coating samples were subjected to 24 h of water leaching prior to JIS Z2801 tests, the Quat compounds on dry coating surfaces were washed away due to their high water solubility. U.S. Patent 9,131,683 B2 demonstrated that the electrostatic attraction between cationic Quat and anionic latex particle can lead to grit formation.[6] Thus, in the dry film of prototypical paint, the Quat cations in the bulk interact strongly with latex particles and cannot migrate to the surface in a sufficient quantity to replenish the depleted Quat at the surface. Therefore, DDAB/DDAP Quat-treated paints were removed from the following EPA residual efficacy and other film property tests.

TABLE 2
Antibacterial Efficacy of iCopper-treated Paints per JIS Z2801

Cu Level	*Staphylococcus aureus* (ATCC 6538; gram positive)			
ppm	Bacteria Count (cfu)	Log10	Log10 Reduction	% Reduction
0	9.9E+04	5.00		
165	6.3E+01	1.80	3.20	99.936
330	<2.0E+01	<1.30	≥3.70	≥99.980
824	<4.0E+01	<1.60	≥3.40	≥99.960
1650	<1.0E+01	<1.00	≥4.00	≥99.990
Cu Level	***Pseudomonas aeruginosa* (ATCC 15442; gram negative)**			
ppm	Bacteria Count (cfu)	Log10	Log10 Reduction	% Reduction
0	1.9E+04	4.28		
165	1.2E+03	3.08	1.20	93.632
330	1.3E+03	3.13	1.15	92.912
824	<4.7E+01	<1.67	≥2.61	≥99.754
1650	2.7E+01	1.43	2.85	99.860

Residual Antibacterial Efficacy per EPA Interim Method

The antibacterial efficacies of iCopper-treated paints were further evaluated using the EPA interim method, which incubated the dry film with the bacterial inoculum at 22 °C and 30-40% RH for 2 h (the film surface was dried out during the incubation). In comparison, the incubation of JIS Z2801 method was maintained at 35 °C and 90% RH throughout the incubation period by trapping the media between the film cover and coating surface. The presence of the wet inoculum media in the JIS Z2801 method promotes the growth of more metabolically active bacteria that are easier to be inhibited or killed than the metabolically dormant bacteria present in the EPA method. Therefore, it is expected that the EPA interim method is more difficult to pass than the JIS Z2801 test.

The residual antibacterial efficacy of the iCopper technology was evaluated in a commercial matte wall paint. As shown in **Table 4,** the iCopper technology enabled > 99.99% or 4 Log10 reduction of *Staphylococcus aureus* and *Pseudomonas aeruginosa* at 1670 ppm Cu after 2 h of contact time. Upon dry abrasion (10 cycles × 16 single passes/cycle) and Quat/NaOCl abrasion (each for 10 cycles × 8 single passes/cycle) that is equivalent to 1-week residual efficacy, the antibacterial efficacy was still maintained at > 99.99% or 4 Log10 reduction. Therefore, the iCopper technology enabled an EPA residual efficacy claim for hygienic paints and coatings.

Since the EPA interim guidance only requires > 99.9% or 3 Log10 reduction for "Supplemental Residual Antimicrobial Products" such as paints and coatings, it is possible to reduce the dose level of the iCopper product from 1670 ppm Cu (> 99.99% or 4 Log10 reduction) to ~850 ppm Cu, as suggested by the JIS Z2801 results (**Table 3**). Furthermore, the EPA test results suggest that the iCopper technology has the potential to achieve the EPA "Residual Disinfectants" claim (≥ 5 Log10 reduction of bacteria within 10 min of contact time). Therefore, more work is planned to further explore the iCopper technology for lower dose levels and the residual disinfectant claim.

Mitigation of Color Impact via Optimization

Most copper compounds are either red (Cu(I) compound) or green-to-blue (Cu(II) compound) and can bring that color into the dry paint film, which is not welcomed by paint companies. As shown in **Table 5,** the iCopper technology does lead to ΔE/ΔE' = 2.54/2.33 at 1150 ppm Cu, which is visually perceivable (ΔE/ΔE' > 2). However, the iCopper technology can be further optimized to mitigate its color impact. The resulting optimized iCopper

TABLE 3
Antibacterial Efficacy of DDAP/DDAB Quat-treated Paints per JIS Z2801

Quat Type	Level (ppm)	*Staphylococcus aureus (ATCC 6538; gram positive)*			
		Bacteria Count (cfu)	Log10	Log10 Reduction	% Reduction
None	0	1.0E+05	5.00		
DDAB	2000	2.4E+04	4.37	0.63	76.412
DDAP	2000	4.2E+04	4.63	0.37	57.807
Quat Type	**Level (ppm)**	***Pseudomonas aeruginosa (ATCC 15442; gram negative)***			
		Bacteria Count (cfu)	Log10	Log10 Reduction	% Reduction
None	0	8.8E+04	4.94		
DDAB	2000	4.9E+04	4.69	0.25	43.939
DDAP	2000	5.7E+04	4.75	0.19	35.606

TABLE 4
Residual Antibacterial Efficacy of iCopper-treated Paints per EPA Interim Method

Sample	Cu Level (ppm)	*Staphylococcus aureus (ATCC 6538; gram positive)*			
		Bacteria Count (cfm)	Log10	Log10 Reduction	% Reduction
Uncoated	0	1.0E+5	5.00		
No abrasion	1670	1.0E+0	0.00	5.00	99.9990%
Dry abraded	1670	1.0E+0	0.00	5.00	99.9990%
Quat abraded	1670	1.0E+0	0.00	5.00	99.9990%
NaOCl abraded	1670	1.0E+0	0.00	5.00	99.9990%
Sample	**Cu Level (ppm)**	***Pseudomonas aeruginosa (ATCC 15442; gram negative)***			
		Bacteria Count (cfm)	Log10	Log10 Reduction	% Reduction
Uncoated	0	2.8E+4	4.44		
No abrasion	1670	1.0E+0	0.00	4.44	99.9964%
Dry abraded	1670	1.0E+0	0.00	4.44	99.9964%
Quat abraded	1670	1.0E+0	0.00	4.44	99.9964%
NaOCl abraded	1670	3.5E+0	0.54	3.90	99.9875%

TABLE 5
Mitigation of Color Impact in iCopper-treated Paints

Sample Type	Cu (ppm)	L	a	b	ΔL	Δa	Δb	ΔE	ΔE'
Control	0	97.16	-0.80	1.94					
iCopper technology	1150	96.14	-2.93	1.00	-1.02	-2.13	-0.94	2.54	2.33
Optimized iCopper technology	1150	95.63	-0.84	1.77	-1.66	-0.06	-0.38	1.70	0.38

The EPA test results suggest that the iCopper technology has the potential to achieve the EPA "Residual Disinfectants" claim (≥ 5 Log10 reduction of bacteria within 10 min of contact time).

Overall, the iCopper technology can contribute to a more hygienic environment.

TABLE 6
Scrub Resistance of iCopper-treated Paints

	Left: Control vs Right: Sample			Left: Sample vs Right: Control				
Cu Level (ppm)	**Control (Cycle #)**	**Sample (Cycle #)**	**(%)**	**Sample (Cycle #)**	**Control (Cycle #)**	**(%)**	**Average (%)**	**Std (%)**
1130	1276	1233	97%	1212	1318	92%	94%	3%
2850	1168	974	83%	1225	1200	102%	93%	13%

FIGURE 3
Scrub resistance of iCopper-treated paints.

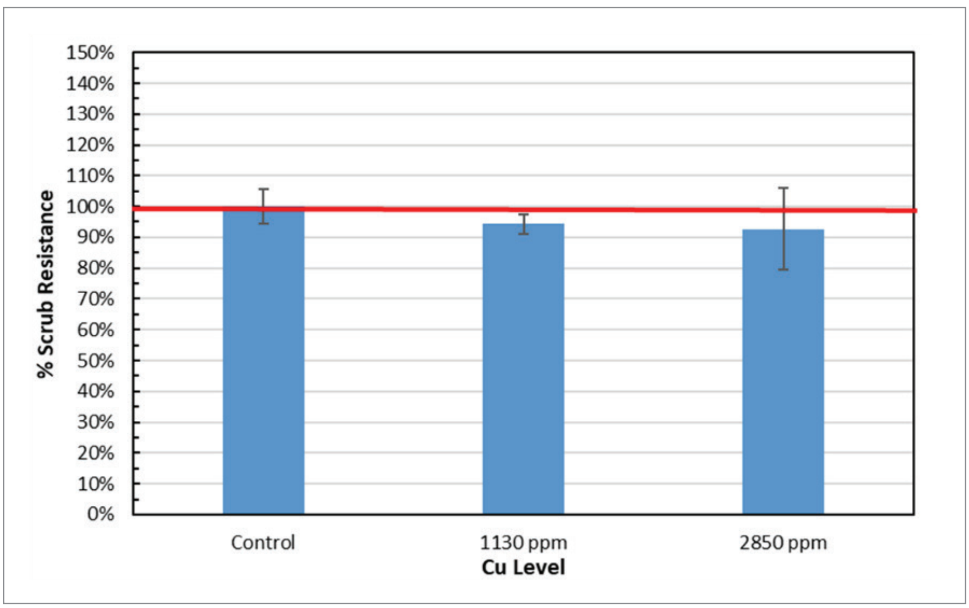
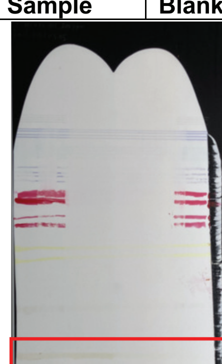
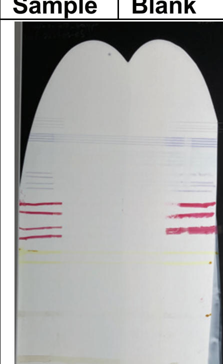


TABLE 7
Stain Resistance of iCopper-treated Paints

Stain Type	1130 ppm Cu	1130 ppm Cu Sample | Blank	2850 ppm Cu	2850 ppm Cu Sample | Blank
Pencil	(=)		(=)	
Pen	(=)		(=)	
Blue highlighter	(=)		(=)	
Purple crayon	(=)		(=)	
305 Lipstick	(=)		(=)	
740 Lipstick	(=)		(=)	
Mustard	(=)		(=)	
Ketchup	(=)		(=)	
Grape juice	(=)		(=)	
Gravy	(=)		(=)	
Coffee	(-)		(-/--)	

technology has reduced ΔE/ΔE' to 1.70/0.38 at the same 1150 ppm Cu, which is no longer visually perceivable. It is worth mentioning that optimization can potentially further enhance the antimicrobial efficacy.

Dry Film Properties: Scrub and Stain Resistance

The impacts of the iCopper technology on dry film properties were assessed in terms of scrub and stain resistance. As shown in **Table 6** and **Figure 3,** the iCopper technology gives < 10% reduction on scrub resistance in a commercial semi-gloss paint at as high as 2850 ppm Cu. The difference is minimal and not statistically significant per standard deviation.

The iCopper technology gives maintained stain resistance in the same commercial semi-gloss paint up to 2850 ppm Cu, except for coffee resistance (**Table** 7). This can be because the iCopper technology is not surface-active, and thus will not enrich on the dry film surface to impact stain resistance.

Conclusions

The iCopper technology has enabled the EPA "Supplementary Residual Antimicrobial Claim" for paints/coatings and further demonstrated its potential to achieve the EPA residual disinfectant claim. Unlike other colorful copper products, its impact on paint color can be mitigated by formulation optimization. The iCopper technology has no negative impact on other dry film properties such as scrub and stain resistance. Overall, the iCopper technology can contribute to a more hygienic environment. Conversely, the hydrophilic-modified DDAP/DDAB Quat products are significantly less efficacious than the iCopper technology upon direct addition into the latex paints and thus are not preferred. ❊

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