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Bioinspired Surface Treatments for Improved Decontamination: Lubricated Paint

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14. ABSTRACT

This effort evaluates bioinspired coatings for use in a top-coat type application to identify those technologies that may improve decontamination capabilities for painted surfaces. This report details results for evaluation of lubricating oils on paint surfaces. Retention of the simulants paraoxon, methyl salicylate, dimethyl methylphosphate, and diisopropyl fluorophosphates following treatment of contaminated surfaces with a soapy water solution is reported. Wetting behaviors and target droplet diffusion on the surfaces are also discussed.

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EXECUTIVE SUMMARY

The Center for Bio/Molecular Science and Engineering at the Naval Research Laboratory (NRL) initiated a program in January 2015 for evaluation of bioinspired treatments suitable for use as a top coat on painted surfaces with the intention of achieving improved aqueous decontamination of these materials. Funding was provided by the Defense Threat Reduction Agency (DTRA, CB10125). Prior reports detailed results for evaluation slippery liquid infused porous surfaces (SLIPS). The controls for these evaluations included lubrication of the paint with oils, producing some reduction in target retention. The current report looks at oils with varied physical characteristics to assess their impact on performance. Retention of the simulants paraoxon, methyl salicylate, dimethyl methylphosphate, and diisopropyl fluorophosphate following treatment of contaminated surfaces with a soapy water solution. Some data on droplet diffusion on the surfaces and wetting angles is also provided.

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BIOINSPIRED SURFACE TREATMENTS FOR IMPROVED DECONTAMINATION: LUBRICATED PAINT

INTRODUCTION

The DoD Chemical and Biological Defense Program (CBDP) seeks to provide protection of forces in a contaminated environment including contamination avoidance, individual protection, collective protection, and decontamination. In January 2015, the Center for Bio/Molecular Science and Engineering at the Naval Research Laboratory (NRL) began an effort funded through the Defense Threat Reduction Agency (DTRA, CB10125) intended to evaluate top-coat type treatments suitable for application to painted surfaces. The intention was to reduce chemical threat agent retention following standard decontamination approaches. The effort sought to survey relevant and related areas of research and evaluate identified technologies under appropriate methods to determine efficacy, scalability, and durability. Slippery liquid-infused porous surfaces (SLIPS) were identified as one potentially valuable technology.[1-4] These coatings comprise a film of lubricating liquid with a textured substrate (micro/nano or both).[5-7] The lubricating liquids were evaluated independently of the full treatments as a point of comparison. During those evaluations the lubricants were noted to reduce target retention. The current document summarizes results for a more diverse set of lubricating oils. The intention of the study was to identify aspects of the oils that contribute to the noted behaviors. Molecular structures of the oils are provided in Figure 1; Table 1 provides details and physical characteristics.

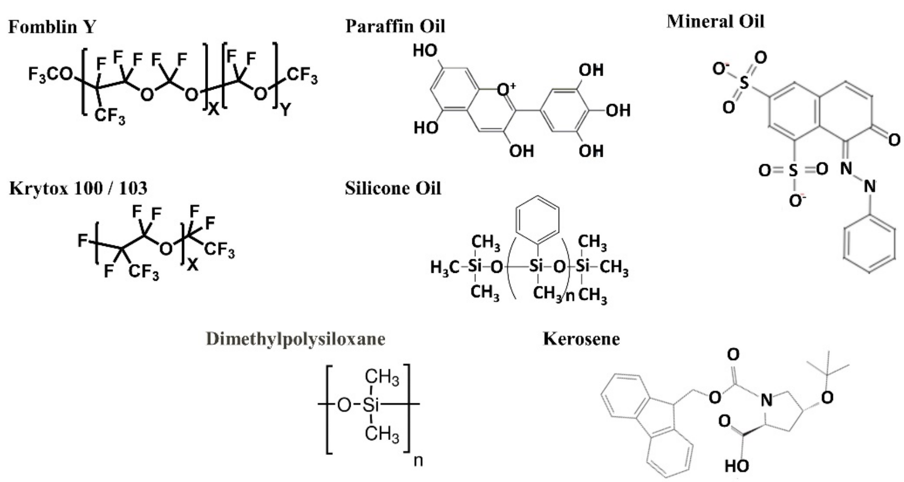

Fig. 1 — Lubricants considered under this study include fluorinated oils (Fomblin Y, Krytox 100, Krytox 103), silicone oils (AR20, AR100, AR200), dimethylpolysilane oils (5, 20, 50, 200, 500 cST @ 20°C), paraffin oil, kerosene, and mineral oils (common, RTM1, RTM3, RTM5).

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Table 1 – Lubricants and Properties

Lubricant	CAS	Density (g/mL)	Viscosity (cST, 20°C)
Fluorinated Oils			
Fomblin Y	69991-67-9	1.88	60
Krytox 100	60164-54-4	1.87	12.4
Krytox 103	60164-54-4	1.92	82
Silicone Oils			
Silicone Oil AR20	63148-58-3	1.01	20
Silicone Oil AP100	63148-58-3	1.06	100
Silicone Oil AR200	63148-58-3	1.05	200
Dimethylpolysiloxane			
DMPS-V	9016-00-6	0.98	5
DMPS-2X	9016-00-6	0.98	20
DMPS-5X	9016-00-6	0.98	50
DMPS-2C	9016-00-6	0.98	200
DMPS-5C	9016-00-6	0.98	500
Other Compounds			
Kerosene	8008-20-6	0.80	2.71
Mineral Oil	8042-47-5	0.84	10
Paraffin Oil	8012-95-1	0.86	100 - 145
Mineral Oil RTM 1	110-54-3	0.66	0.31
Mineral Oil RTM 3	8042-47-5	0.84	0.95
Mineral Oil RTM 5	64742-47-8 / 8042-47-5	0.88	3.33

METHODS

Sessile contact angles for samples evaluated under this effort used three 3 μL droplets per surface with each droplet measured independently three times for each of three targets, water, ethylene glycol, and n-heptane. Geometric surface energy was calculated based on the water and ethylene glycol interactions using software designed for the DROPimage goniometer package. Sliding angles were determined using 5 μL droplets. The droplet was applied at 0° after which the supporting platform angle was gradually increased up to 60°. Sliding angles for each of the liquids were identified as the angle for which movement of the droplet was identified. Shedding angles for each liquid were determined using 12 μL droplets initiated 2.5 cm above the coupon surface. Changes in base angle of 10° were utilized to identify the range of droplet shedding angle based on a complete lack of droplet retention by the surface (not sliding). The angle was then reduced in steps of 1° to identify the minimum required angle. Droplet diameters were determined using tools provided by Adobe Photoshop CS3. Droplets of 5 μL were applied to the surfaces and images were collected at 30 s intervals for 5 min followed by images at 5 min intervals for a total of 30 min. DFP samples were kept covered for the duration of the experiment to minimize evaporation. In some cases, reflections from the glass cover can be seen in the images.

Simulant exposure and evaluation methods were based on the tests developed by Edgewood Chemical Biological Center referred to as Chemical Agent Resistance Method (CARM).[8] Standard target exposures utilized a challenge level of 10 g/m^2. Here, the coupons were 0.00101 m^2; a 10 g/m^2 target challenge was applied to the surfaces as two equally sized neat droplets. Following application of the target, coupons were aged 1 h. Decontamination used a gentle stream of air to expel target from the surface prior to rising with soapy water (0.59 g/L Alconox in deionized water). The coupons were then soaked in isopropanol for 30 min to extract remaining target; this isopropanol extract was analyzed by the appropriate chromatography method to determine target retention on the surface.

For target analysis, gas chromatography-mass spectrometry (GC-MS) was accomplished using a Shimadzu GCMS-QP2010 with AOC-20 auto-injector equipped with a Restex Rtx-5 (30 m x 0.25 mm ID x 0.25 μm df) cross bond 5% diphenyl 95% dimethyl polysiloxane column. A GC injection temperature of 200°C was used with a 1:1 split ratio at a flow rate of 3.6 mL/min at 69.4 kPa. The oven gradient ramped from 50°C (1 min hold time) to 180°C at 15°C/min and then to 300°C at 20°C/min where it was held for 5 min.

RESULTS

When the soapy water process (CARM) was employed for paint only coupons, retention was significant but was less than that of paint only coupons that were extracted with no rinsing or decontamination steps. For comparison purposes, paint only coupons that were not rinsed prior to isopropanol extraction retained the following: paraoxon – 9.84 g/m^2, MES – 9.54 g/m^2, DMMP – 9.90 g/m^2, DFP - 7.39 g/m^2. Though the nominal target application was 10 g/m^2, recovery from surfaces was always less than this value. Losses due to evaporation would be expected, especially for DFP. Additional losses likely occur during rinse steps due to agent interaction with the untreated region of the coupon; the back of these coupons is unpainted aluminum.

The CARM process was used to evaluate painted surfaces lubricated with each of the considered oils (Figure 2; Table 2), retention of all targets was similarly reduced for fluoropolymer treated surfaces and for Fomblin Y lubricated surfaces. The impact of lubrication of the paint by Krytox 100 and 103 was also considered. Fomblin Y, Krytox 100, and Krytox 103 are fluorocarbon ether polymers of polyhexafluoropropylene oxide (also known as perfluoropolyether (PFPE), perfluoroalkylether (PFAE) and perfluoropolyalkylether (PFPAE)). The oils are stable, non-flammable, nonvolatile (to >150°F), and insoluble in water, acid, base, and most organic solvents. Fomblin Y has molecular formula $CF_3O(-CF(CF_2)CF_2O-)_x(-CF_2O-)_yCF_3$ and average molecular weight 1800 with density 1.88 g/cm^3 and viscosity 60 cST. The Krytox oils have molecular formula $F-(CF(CF_3)-CF_2-O)_x-CF_2CF_3$. Krytox 100 has density 1.87 g/cm^3 and viscosity 12.4 cST with average molecular weight approximately 1800 while Krytox 103 has density 1.92 g/cm^3 and viscosity 82 cST with average molecular weight approximately 3500. No dependence between retention and density or viscosity was noted either for lubrication of painted surfaces or for lubrication of a polymer-based slippery liquid infused porous surface treatment (Table 2).[7]

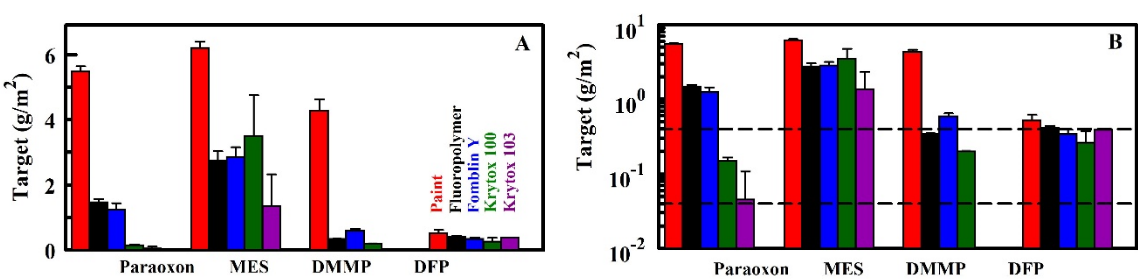

Fig. 2 — Target retention by coupons following treatment with an air stream and rinsing with soapy water: paint (red) and paint with fluoropolymer (black), Fomblin Y (blue), Krytox 100 (green), and Krytox 103 (purple) plotted on a linear (A) and a log scale (B).

Table 2 – Fluorinated Oils: Target Retention (g/m^2) Following 1 h Aging on Aluminum Supports

Coupon	Paraoxon	MES	DMMP	DFP
Controls				
Paint Only	5.48	6.20	4.28	0.52
Smooth Fluoropolymer	1.47	2.74	0.34	0.41
Fluorinated Oils				
Fomblin Y Oiled Paint	1.24	2.85	0.59	0.34
Krytox 100	0.15	3.50	0.20	0.26
Krytox 103	0.04	1.34	ND	0.39
Fluorinated Oils on Polymer SLIPS				
SLIPS with Fomblin Y	2.27	1.26	0.10	0.23
SLIPS with Krytox 100	3.13	4.82	0.23	1.02
SLIPS with Krytox 103	3.33	3.83	0.20	0.46

*ND indicates not detected

A wider variety of silicone (polyphenyl-methylsiloxane) and dimethylpolysiloxane (DMPS) oils with varied viscosities and densities are readily available. The impact of lubricating the paint with a series of these materials was also considered. The CARM process was used to evaluate painted surfaces lubricated with each of the considered silicone oils (Figure 3; Table 3). These materials provided densities of 1.01, 1.06, and 1.05 g/mL with viscosities of 20, 100, and 200 cST (20°C; Table 1). Lubrication of the painted surfaces with these oils reduced retention of the targets. This series appeared to point toward improved performance in oils of lower viscosity.

The second set of materials, dimethylpolysiloxane oils, was selected to provide a larger series of variations in viscosity with fixed density. All DMPS materials provide a density of 0.98 g/mL with viscosities varying from 5 to 500 cST (20°C; Table 1). The results do not support the apparent trend (dependence on viscosity) noticed in the silicone oil series (Figure 4). This series also indicates that there is no dependence on the density of the oil used in the scenario. In addition to the silicone and DMPS oils, a group of common oils (kerosene, paraffin oil, mineral oil) were evaluated. A third series of oils was also considered, mineral oils (Table 3). As shown in Figures 5 and 6, these materials did not provide any additional evidence of a relationship between retention and viscosity or density.

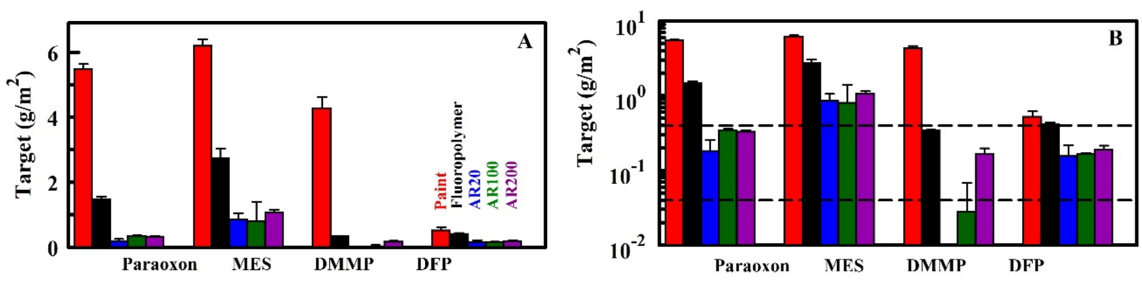

Fig. 3 — Target retention by coupons following treatment with an air stream and rinsing with soapy water: paint (red) and paint with fluoropolymer (black), silicone oil AR20 (blue), silicone oil AR100 (green), and silicone oil AR200 (purple) plotted on a linear (A) and a log scale (B).

Table 3 – Oil Series: Target Retention (g/m^2) Following 1 h Aging on Aluminum Supports

Coupon	Paraoxon	MES	DMMP	DFP
Controls				
Paint Only	5.48	6.20	4.28	0.52
Smooth Fluoropolymer	1.47	2.74	0.34	0.41
Silicone Oils				
Silicone Oil AR20	0.18	0.85	ND	0.16
Silicone Oil AP100	0.35	0.80	0.03	0.17
Silicone Oil AR200	0.33	1.06	0.17	0.19
Dimethylpolysiloxane				
DMPS-V	0.17	1.70	ND	ND
DMPS-2X	0.18	1.56	ND	0.02
DMPS-5X	ND	0.90	ND	0.02
DMPS-5C	ND	2.34	0.25	0.19
DMPS-2C	ND	0.57	0.08	0.10
Other Compounds				
Kerosene	ND	0.69	ND	0.10
Mineral Oil	0.31	0.38	ND	0.43
Paraffin Oil	ND	0.49	0.07	0.19
Mineral Oil RTM1	1.94	5.97	ND	0.19
Mineral Oil RTM3	0.36	3.21	ND	0.22
Mineral Oil RTM5	0.13	1.00	0.08	0.04

*ND indicates not detected

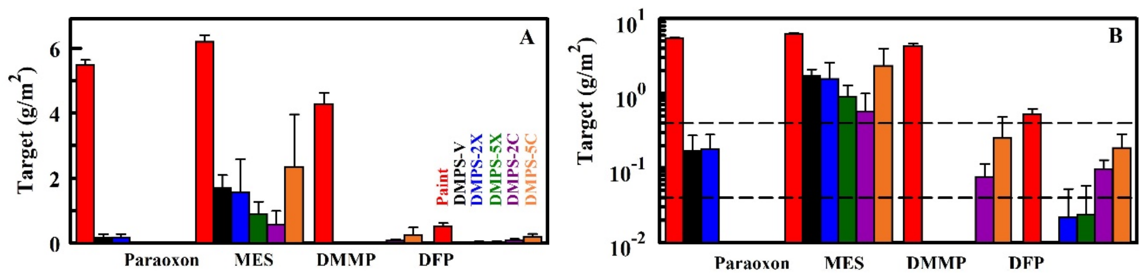

Fig. 4 — Target retention by coupons following treatment with an air stream and rinsing with soapy water: paint (red) and paint with DMPS-V (black), DMPS-2X (blue), DMPS-5X (green), DMPS-2C (purple), and DMPS-5C (orange) plotted on a linear (A) and a log scale (B).

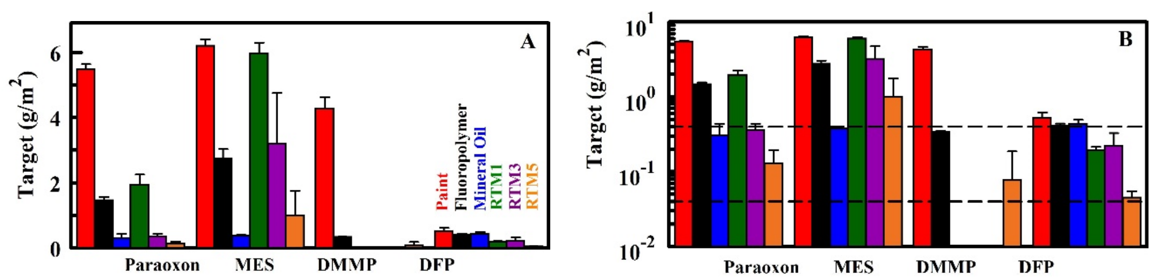

Fig. 5 — Target retention by coupons following treatment with an air stream and rinsing with soapy water: paint (red) and paint with fluoropolymer (black), mineral oil (blue), paraffin oil (green), and kerosene (purple) plotted on a linear (A) and a log scale (B).

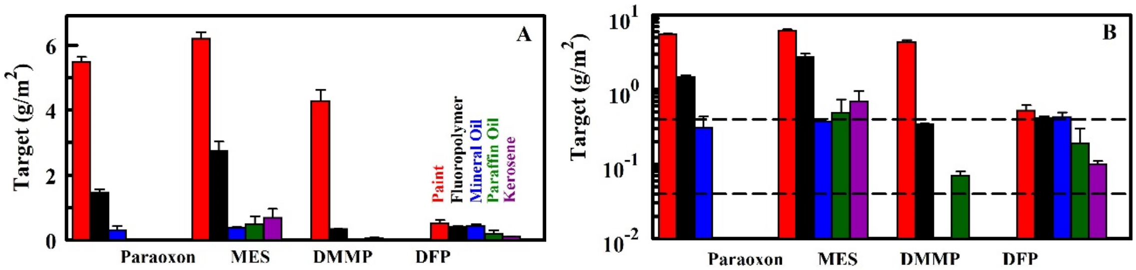

Fig. 6 — Target retention by coupons following treatment with an air stream and rinsing with soapy water: paint (red) and paint with fluoropolymer (black), mineral oil (blue), mineral oil RTM1 (green), mineral oil RTM3 (purple), and mineral oil RTM5 (orange) plotted on a linear (A) and a log scale (B).

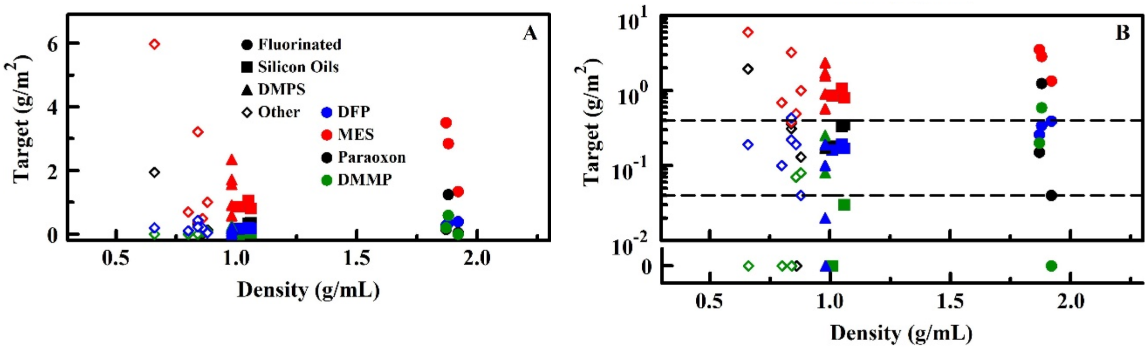

Fig. 7 — Target retention by coupons versus the density of the lubricating oil: paraoxon (black), MES (red), DMMP (green), and DFP (blue). Symbols are divided by oil type: fluorinated oils (circle), silicone oils (square), DMPS (triangle), and other compounds (diamond). Data is plotted on a linear (A) and a log scale (B).

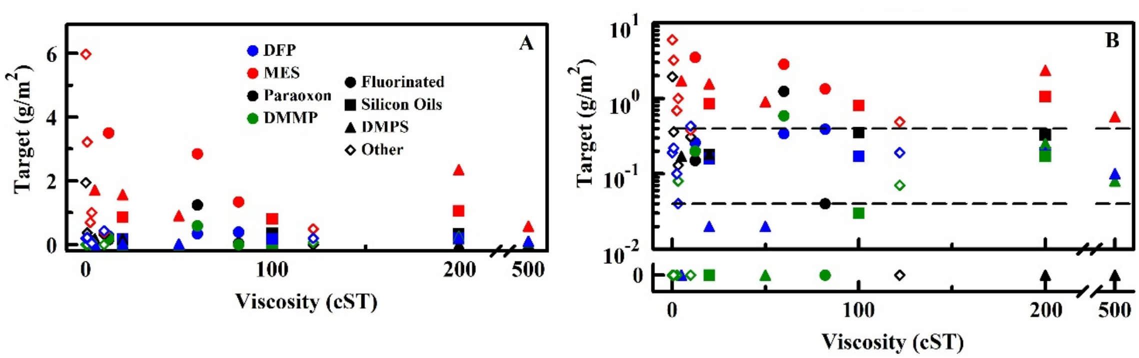

Fig. 8 — Target retention by coupons versus the viscosity of the lubricating oil: paraoxon (black), MES (red), DMMP (green), and DFP (blue). Symbols are divided by oil type: fluorinated oils (circle), silicone oils (square), DMPS (triangle), and other compounds (diamond). Data is plotted on a linear (A) and a log scale (B).

CONCLUSIONS

The reduction in target retention noted for oil lubricated, SLIPS control samples motivated the current study. It is suspected that targets are retained within the oil during exposure and are subsequently removed during the CARM process. While the current data set indicates that retention of targets on painted surfaces can be reduced through application of a sacrificial oil, no relationships between performance and physical properties could be identified. As with many of the treatments evaluated under this effort, lubrication of paint using any of the oils evaluated will produce changes in the appearance of the painted surface (Appendices), lending a wet look to the surfaces. Spectrophotometric analysis is necessary to determine the overall impact on color and reflectivity. Retention levels at 4% and 0.4% of applied target are indicated by dashed lines in the provide graphs. Achieving significant reductions in methyl salicylate retention was a challenge across all of the considered oils. Commonly available paraffin oil, kerosene, and mineral oil reduced retention to the 4% level for all targets. Lubrication of painted surfaces using commonly available oils is unlikely to meet the goals of this effort with respect to improved performance under standard decontamination conditions; however, the potential for some reduction in target retention has been demonstrated.

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Appendix A

WETTING BEHAVIORS

Analysis of the support surface in the absence of lubricating liquid provides a point of comparison for evaluating the benefits of the surface treatment; each table includes data on the relevant support material, a painted aluminum coupon, as well as a smooth fluoropolymer top coat.[7] The Fomblin Y and the fluoropolymer coating reduce the surface energy of the coupons (Table A1). No sliding was noted on any of the evaluated control surfaces below 60°. Shedding angles for the Fomblin Y surface were between 35° and 50° for test liquids. The wetting data provided here does not capture behaviors for the full set of materials considered. Prior work has shown a lack of correlation between wetting behaviors and retention. For this reason, no effort was made to complete this data set.

Table A1 – Sessile, Sliding, and Shedding Contact Angles on Aluminum Supports

Coupon	**Liquid**	**Sessile Angle**	**Sliding Angle**	**Shedding Angle**	**Geometric Surface Energy (mJ/m^2)**
Controls					
Paint Only	water	47.5 ± 1.1	>60	>60	71.9 ± 5.1
	ethylene glycol	55.7 ± 2.1	>60	>60	
	n-heptane	--	--	--	
Fluoropolymer	water	120.6 ± 0.69	>60	>60	18.6 ± 3.1
	ethylene glycol	98.6 ± 2.7	>60	>60	
	n-heptane	21.3 ± 1.2	>60	>60	
Fluorinated Oils					
Fomblin Y Oiled Paint	water	73.1 ± 2.1	>60	46.7 ± 3.3	32.2 ± 1.6
	ethylene glycol	52.5 ± 0.6	>60	49.8 ± 4.9	
	n-heptane	40.1 ± 2.9	>60	36.6 ± 3.3	
Krytox 100	water	64.1 ± 0.3	>60	>60	44.7 ± 1.7
	ethylene glycol	63.8 ± 0.4	>60	>60	
	n-heptane	--	>60	>60	
Silicone Oils					
Silicone Oil AR20	water	83.9 ± 0.8	37.4 ± 2.8	43.7 ± 2.9	24.7 ± 0.7
	ethylene glycol	65.4 ± 0.8	30.2 ± 10.3	29.2 ± 1.7	
	n-heptane	--	>60	>60	
Silicone Oil AR200	water	94.2 ± 0.4	>60	>60	23.5 ± 0.6
	ethylene glycol	70.4 ± 0.4	37.2 ± 4.8	24.3 ± 4.3	
	n-heptane	--	>60	>60	
Dimethylpolysiloxane					
DMPS-2X	water	97.2 ± 0.4	>60	>60	16.2 ± 0.2
	ethylene glycol	79.6 ± 0.2	>60	21.8 ± 5.6	
	n-heptane	--	>60		
DMPS-5C	water	65.4 ± 0.2	>60	>60	38.6 ± 0.5
	ethylene glycol	71.3 ± 0.3	>60	>60	
	n-heptane	--	>60	>60	

The tendency of droplets to spread across the surfaces was also evaluated (Appendices B through I). For these studies, droplets of the simulants (5 μL) were utilized. The spread of the droplets was quantified by measuring the diameter of the droplets in the images over time (Figures A1 and A2). For the paint only samples, MES and DFP spread quickly reaching the edges of the coupon at 10 and 2 min, respectively. DMMP does not spread during the course of the 30 min incubation. Similar behavior is noted for the Fomblin Y oiled coupons. The fluoropolymer coating significantly reduces spread of the three targets on the coupon. The wetting data provided here does not capture behaviors for the full set of materials considered. Prior work has shown a lack of correlation between wetting behaviors and retention. For this reason, no effort was made to complete this data set.

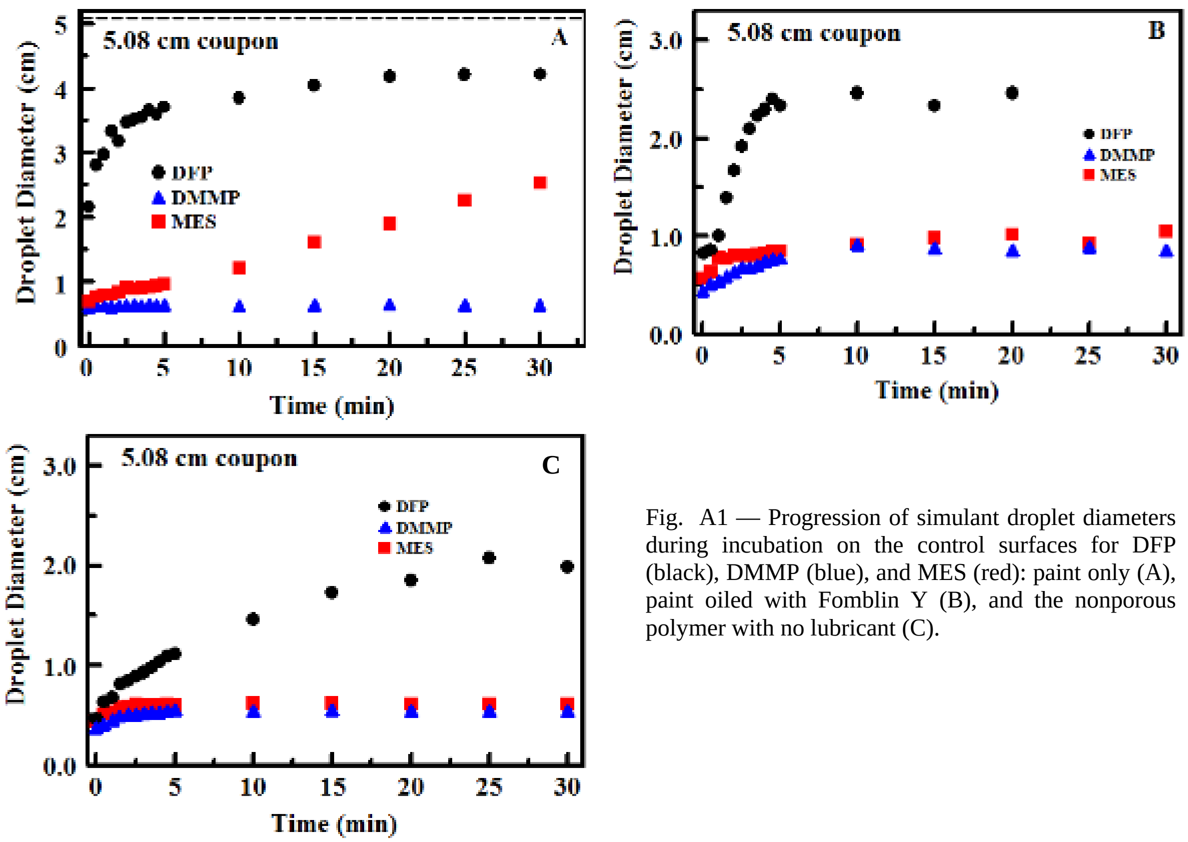


Fig. A1 — Progression of simulant droplet diameters during incubation on the control surfaces for DFP (black), DMMP (blue), and MES (red): paint only (A), paint oiled with Fomblin Y (B), and the nonporous polymer with no lubricant (C).

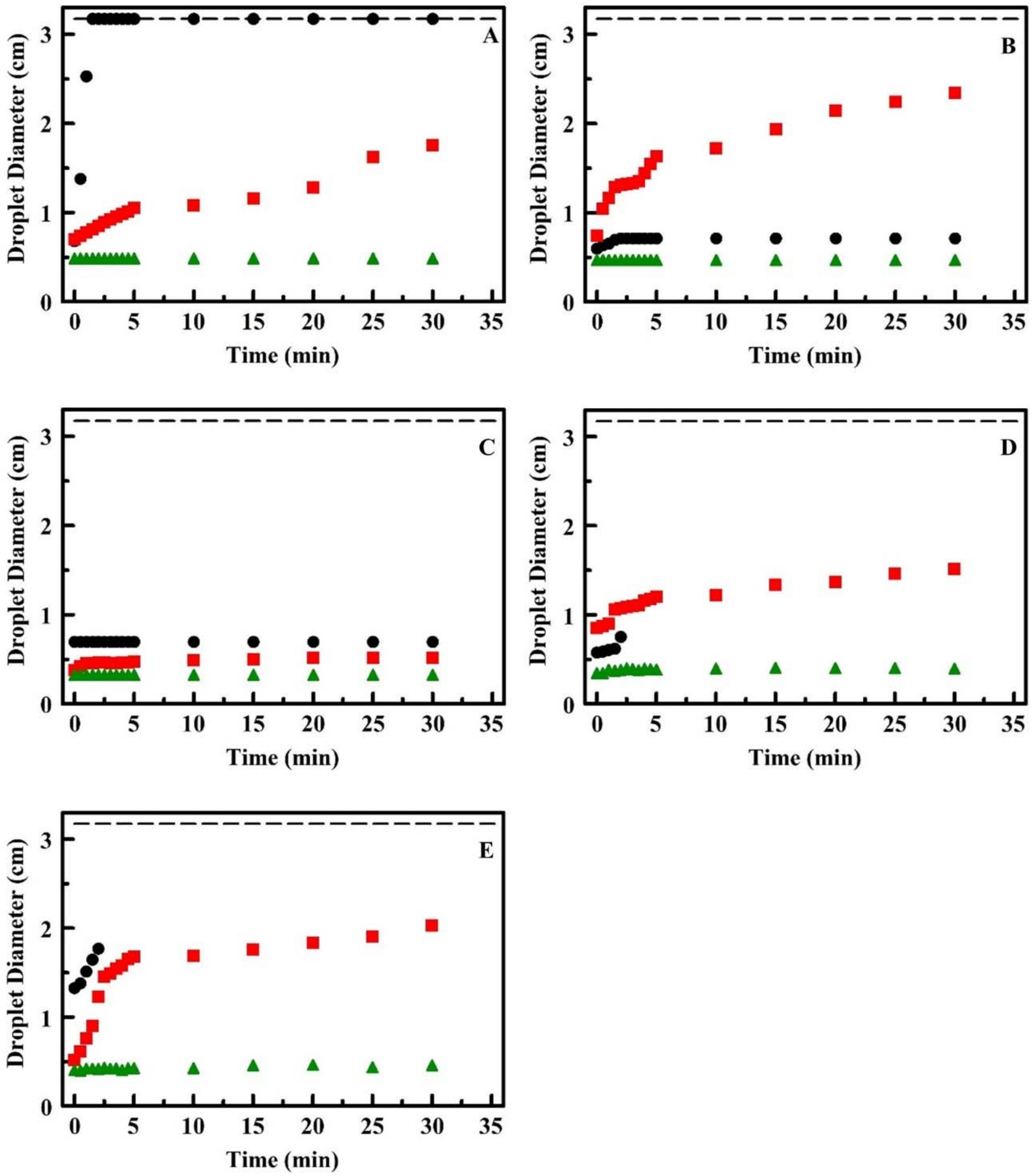


Fig. A2 — Progression of simulant droplet diameters during incubation on the control surfaces for DFP (black), DMMP (green), and MES (red): paint lubricated with silicone oil AR20 (A), with silicone oil AR200 (B), with DMPS 2C (C), with DMPS 5X (D), and with Krytox 100 (E).

Appendix B

PAINTED COUPON IMAGES

Fig. B1 — Paraoxon on paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1.0 (D), 1.5 (E), 2.0 (F), 2.5 (G), 3.0 (H), 3.5 (I), 4.0 (J), 4.5 (K), 5.5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

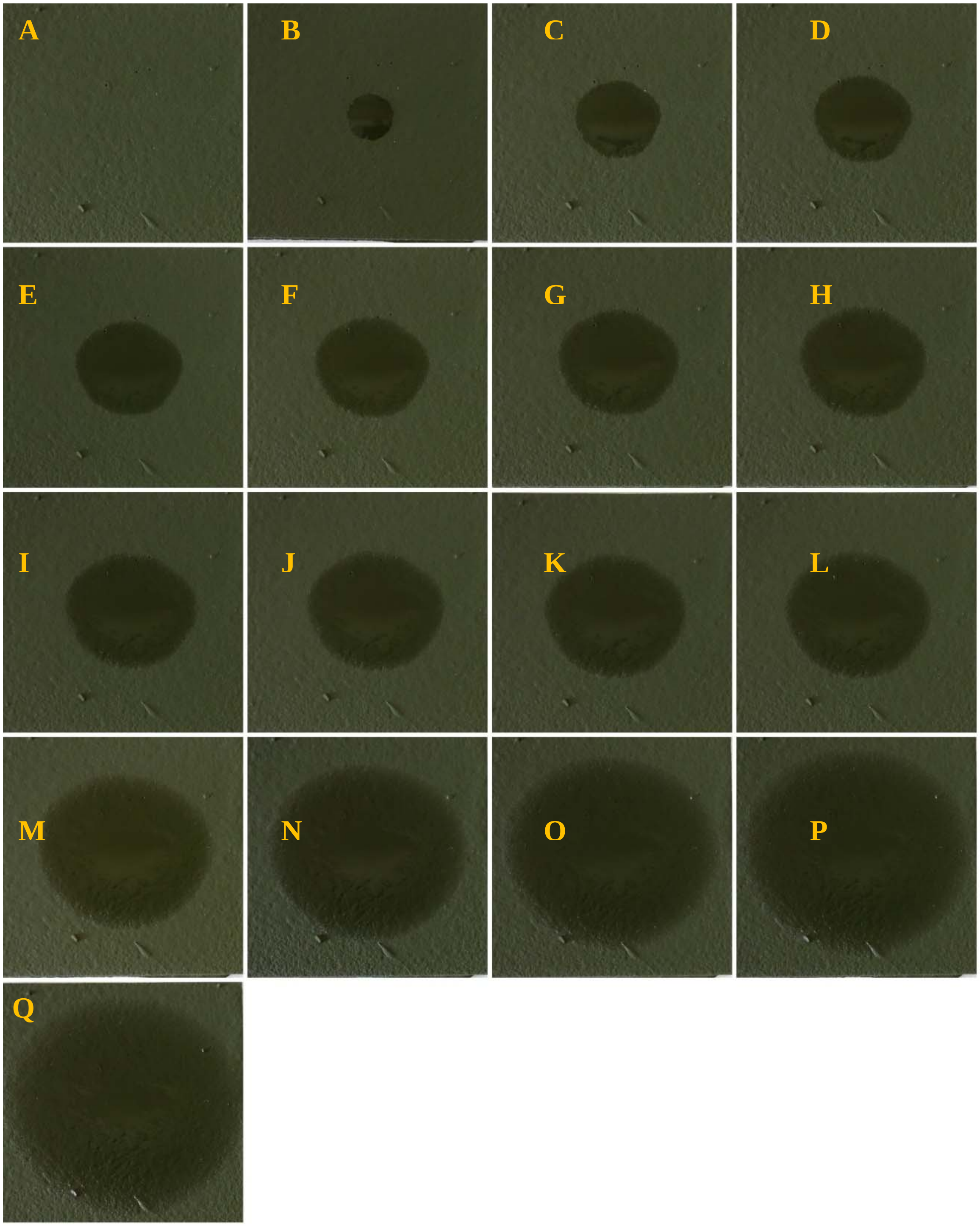


Fig. B2 — MES on paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

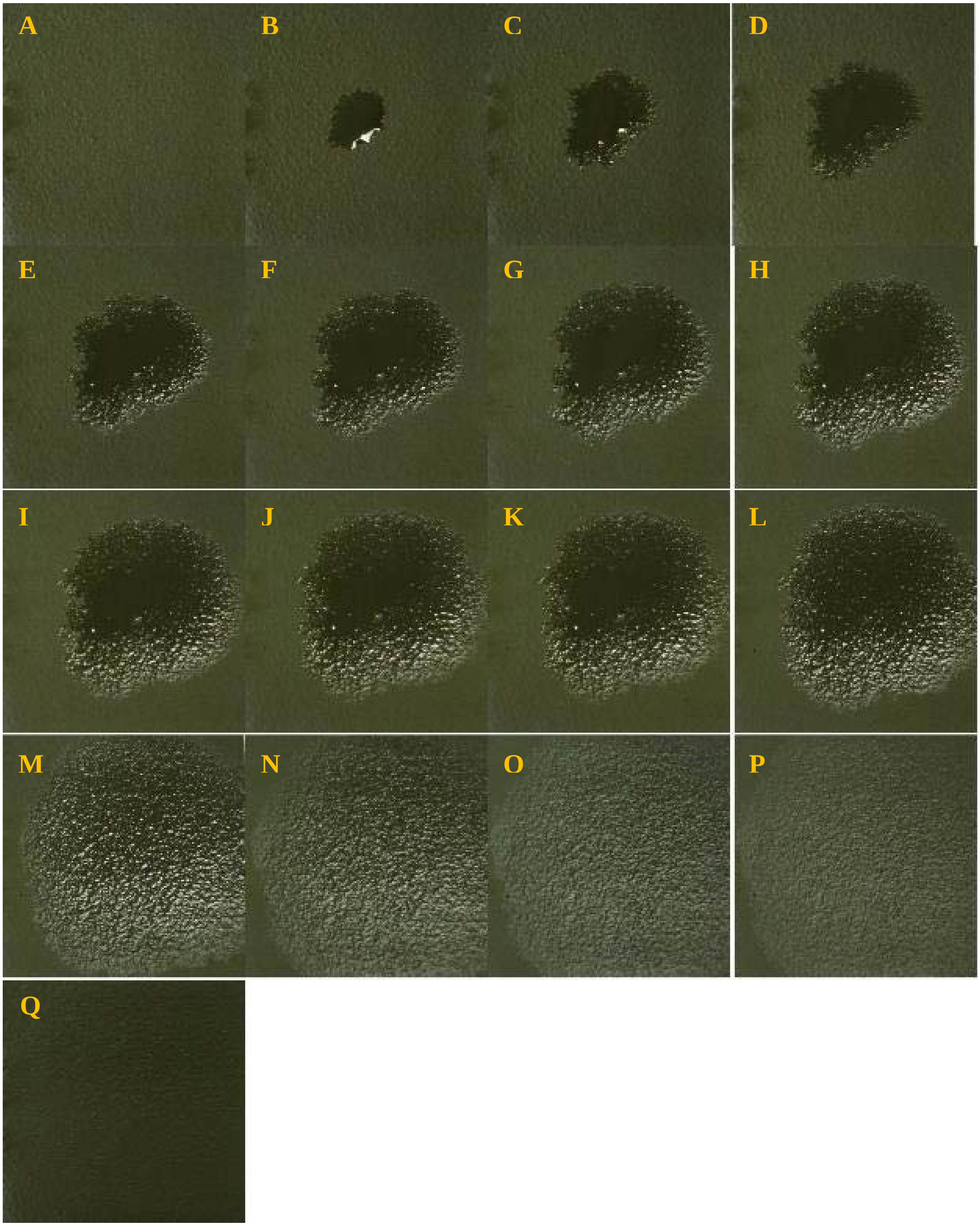
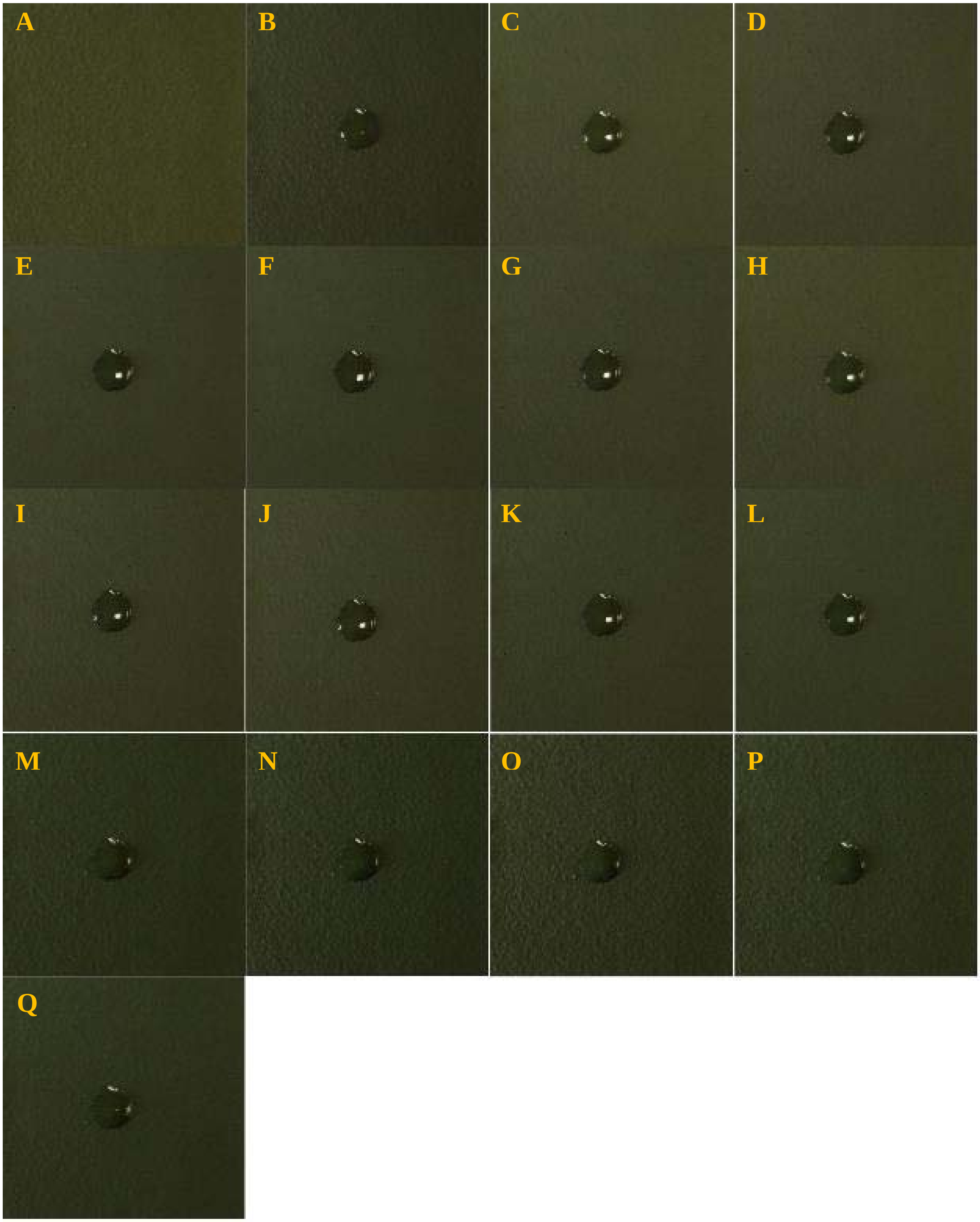


Fig. B3 — DMMP on paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.



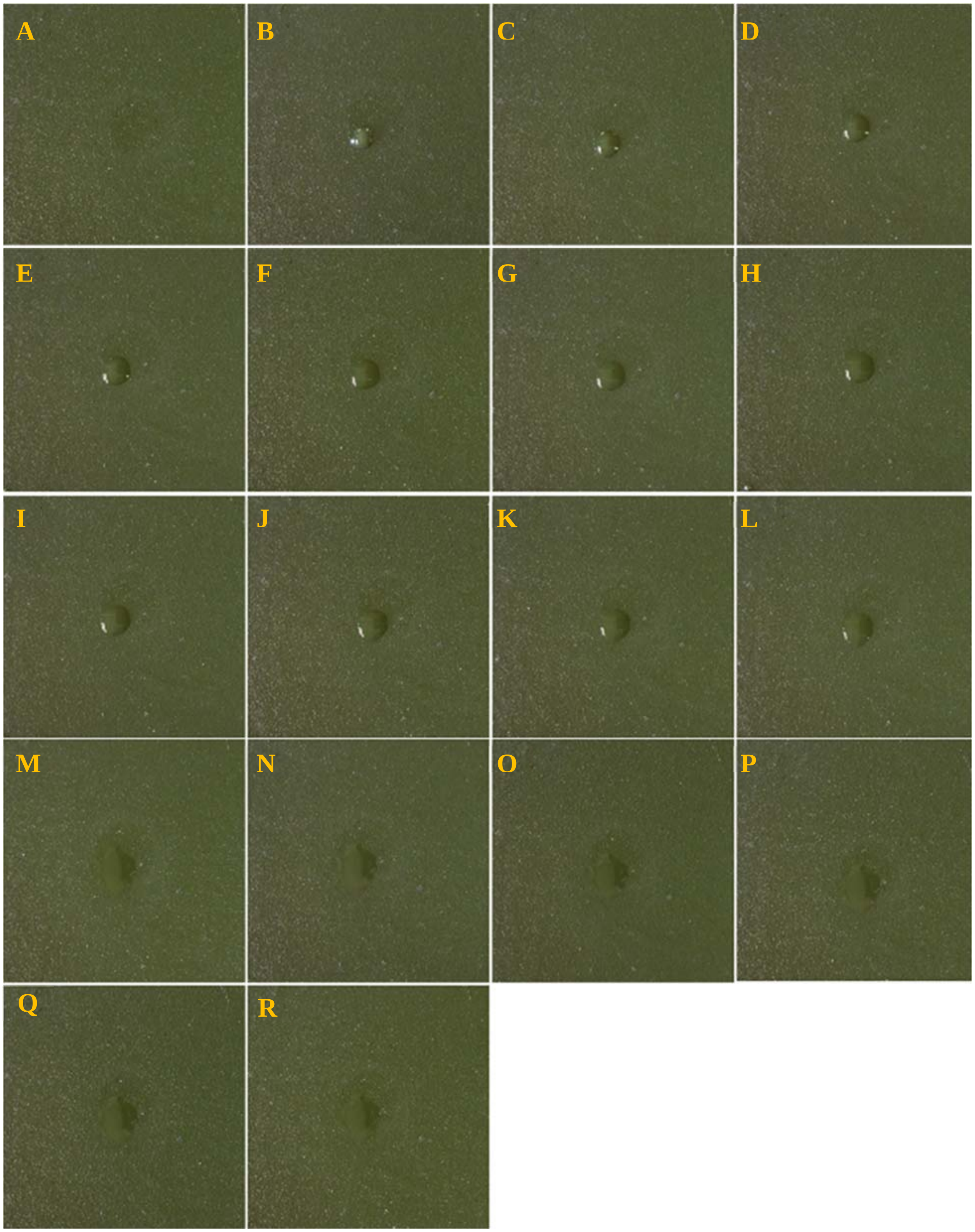
Appendix C

FOMBLIN Y LUBRICATED PAINT IMAGES

Fig. C1 — DFP on Fomblin Y oiled paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 5.5 (M), 10 (N), 15 (O), 20 (P), 25 (Q), and 30 (R) min following application of the target. These images were collected with a glass cover in place to limit evaporation. Reflections from the cover can be seen in some images.

Fig. C2 — MES on Fomblin Y oiled paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 5.5 (M), 10 (N), 15 (O), 20 (P), 25 (Q), and 30 (R) min following application of the target.

Fig. C3 — DMMP on Fomblin Y oiled paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 5.5 (M), 10 (N), 15 (O), 20 (P), 25 (Q), and 30 (R) min) min following application of the target.



Appendix D

SMOOTH POLYMER IMAGES

Fig. D1 — DFP on the nonporous polymer with no lubricant. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 5.5 (M), 10 (N), 15 (O), 20 (P), 25 (Q), and 30 (R) min following application of the target. These images were collected with a glass cover in place to limit evaporation. Reflections from the cover can be seen in some images.

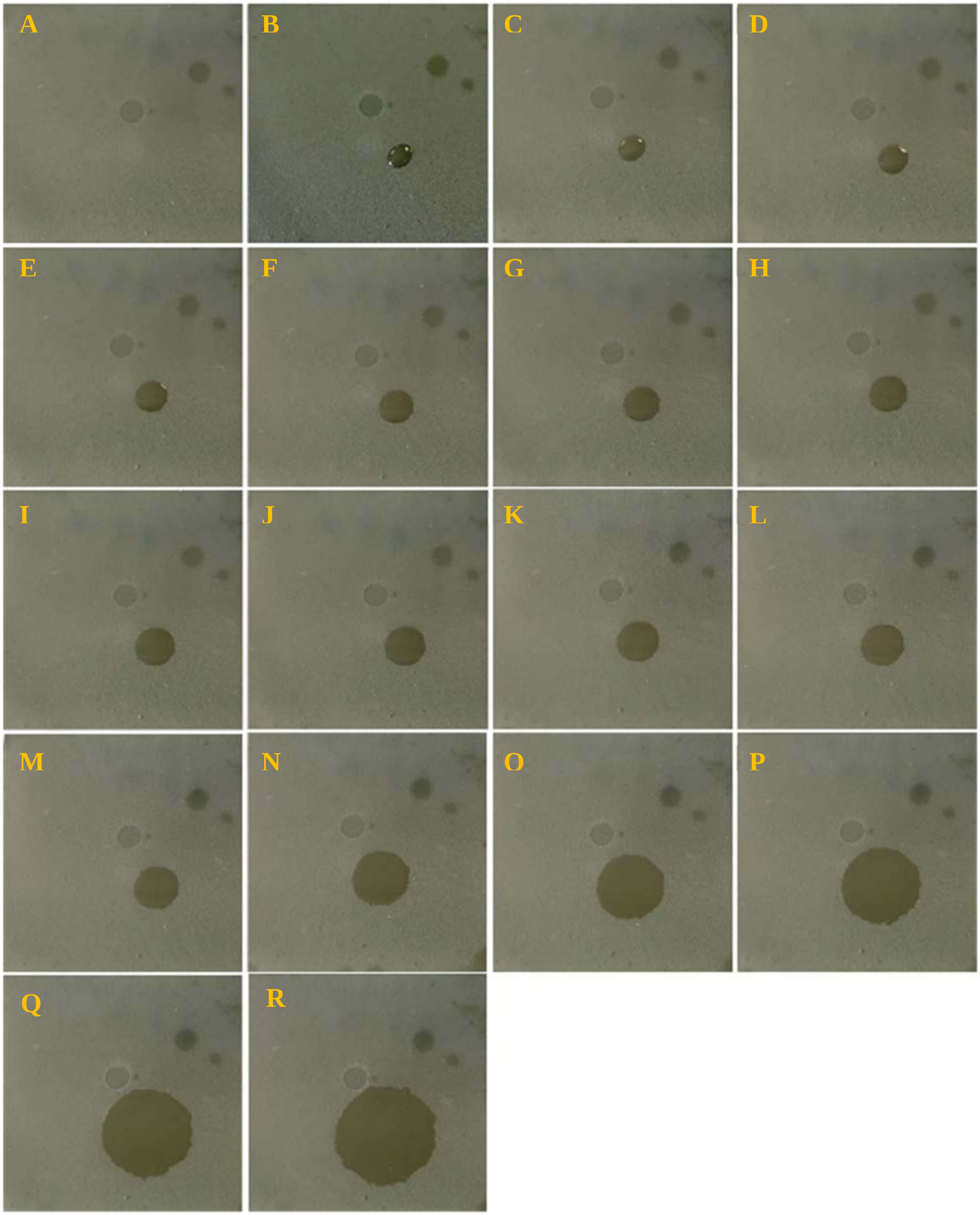


Fig. D2 — MES on the nonporous polymer with no lubricant. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

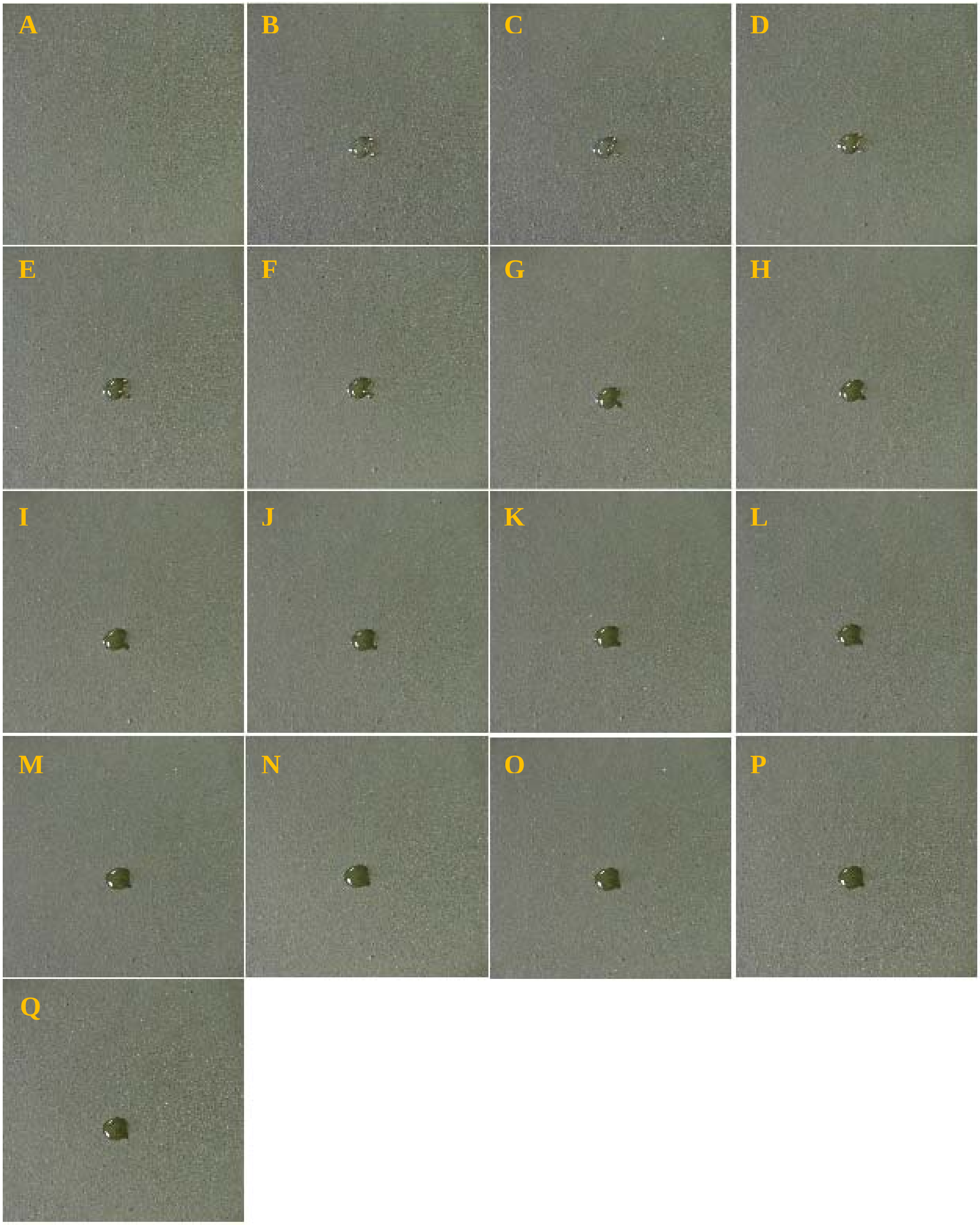
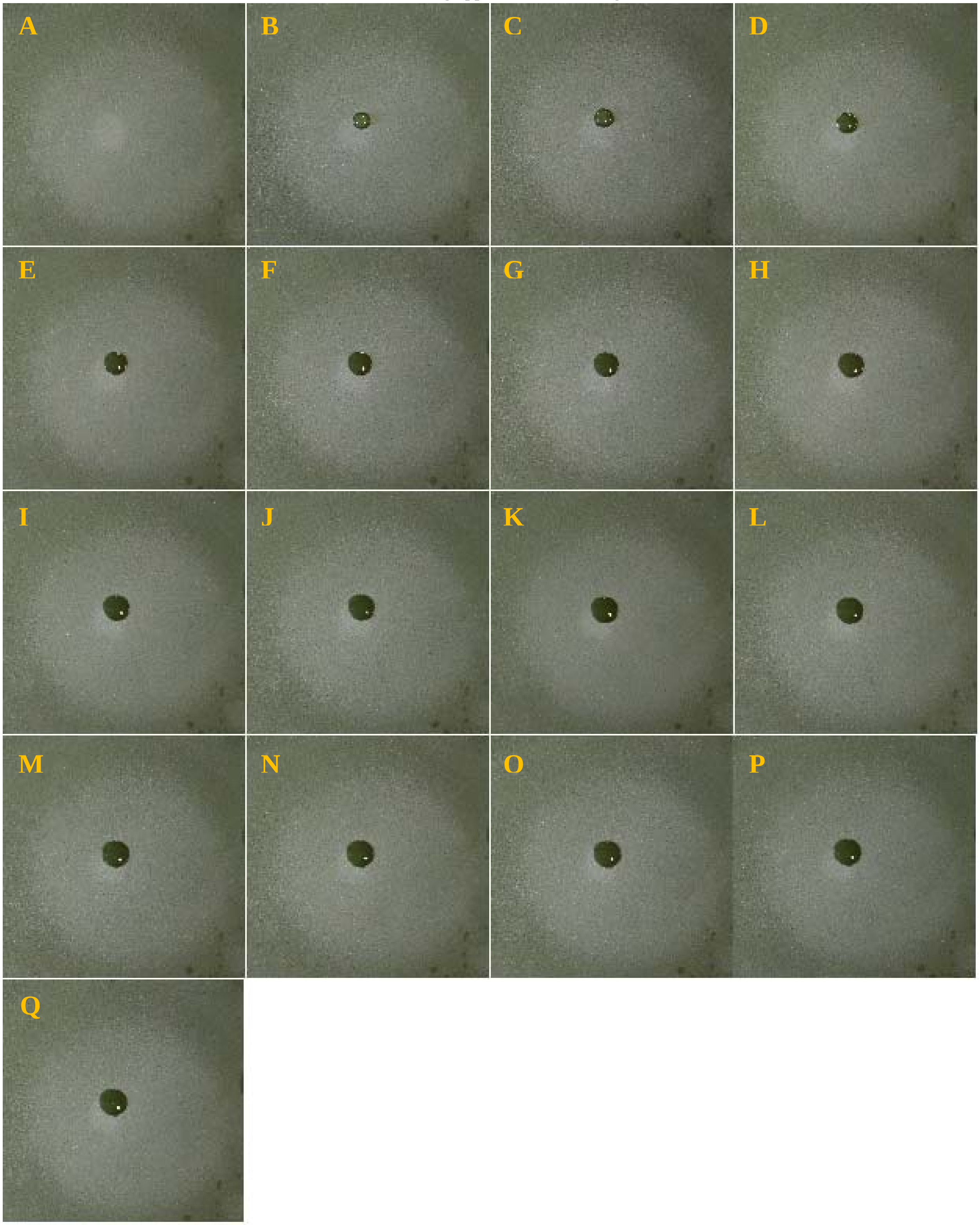


Fig. D3 — DMMP on the nonporous polymer with no lubricant. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.



Appendix E

KRYTOX 100 LUBRICATED PAINT IMAGES

Fig. E1 — DFP on the Krytox 100 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

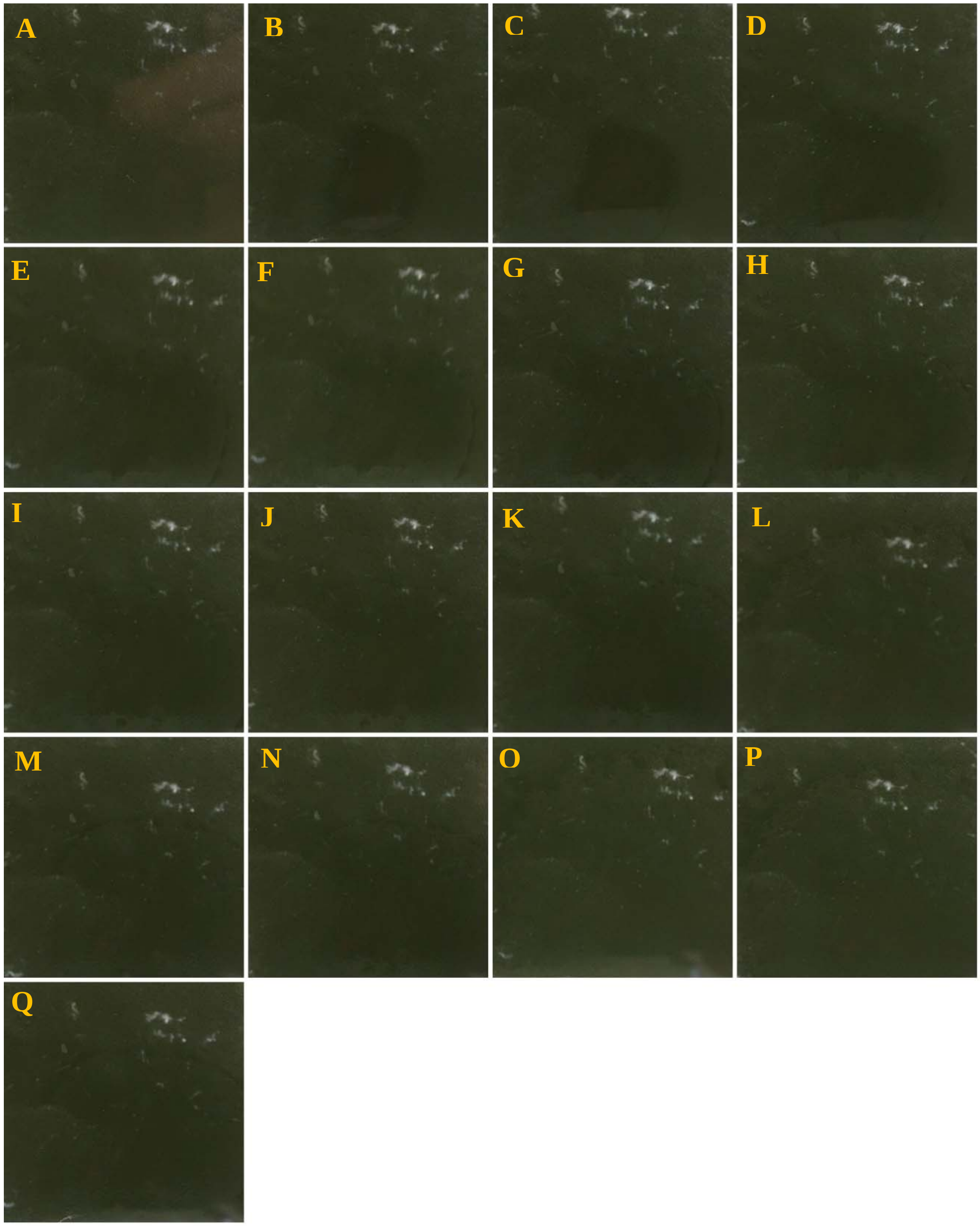


Fig. E2 — MES on the Krytox 100 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

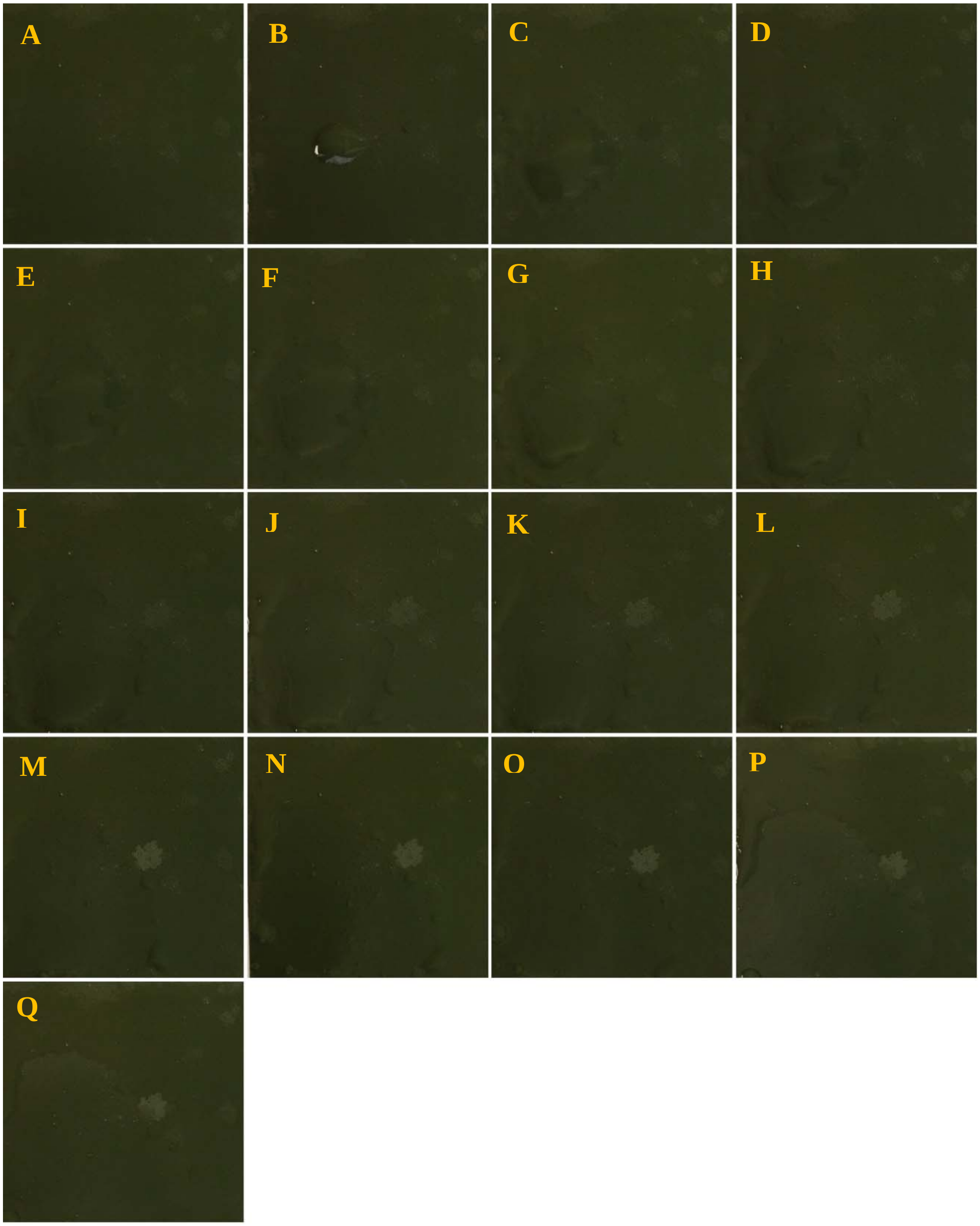
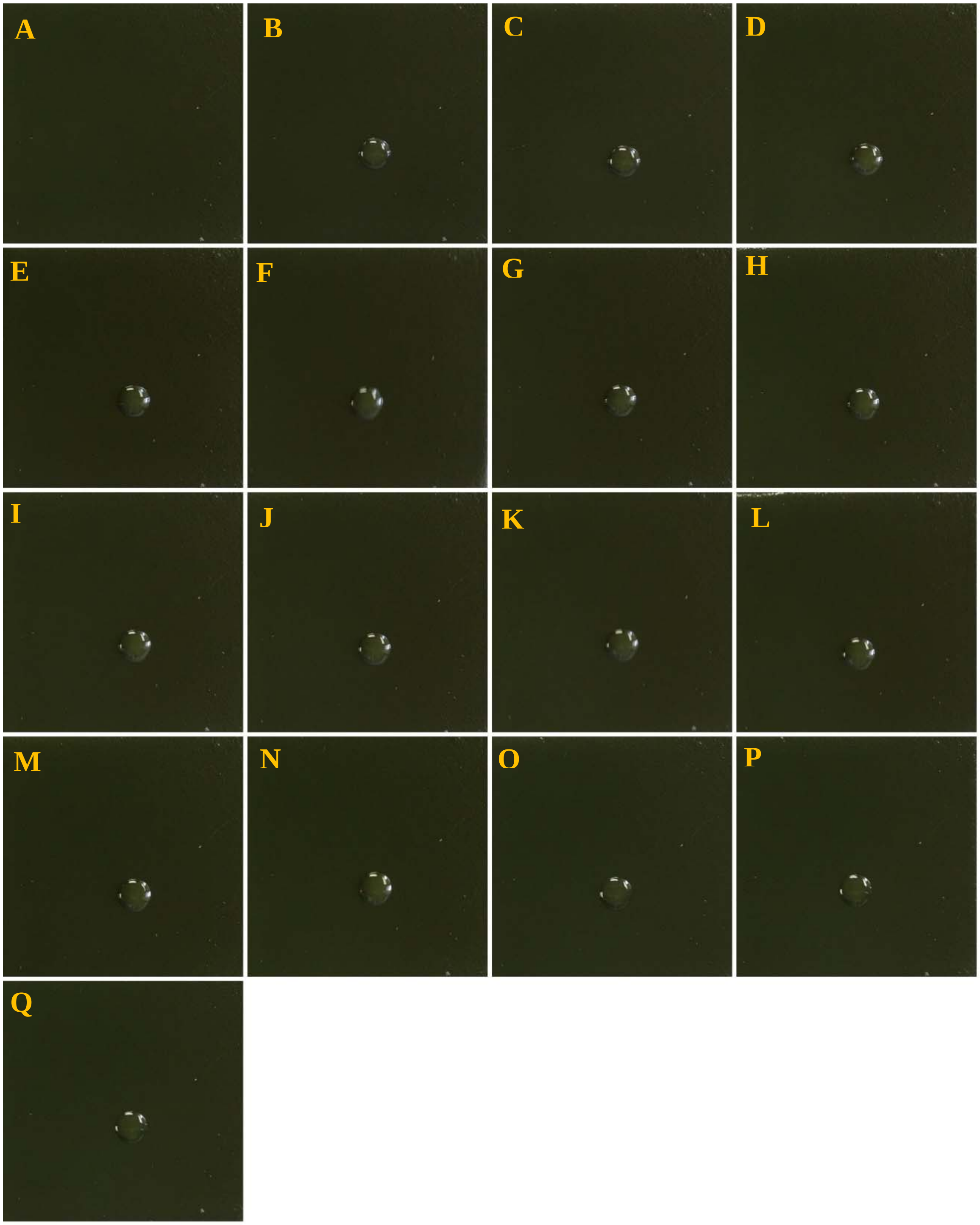


Fig. E3 — DMMP on the Krytox 100 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.



Appendix F

SILICONE OIL AR20 LUBRICATED PAINT IMAGES

Fig. F1 — DFP on the silicone oil AR20 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

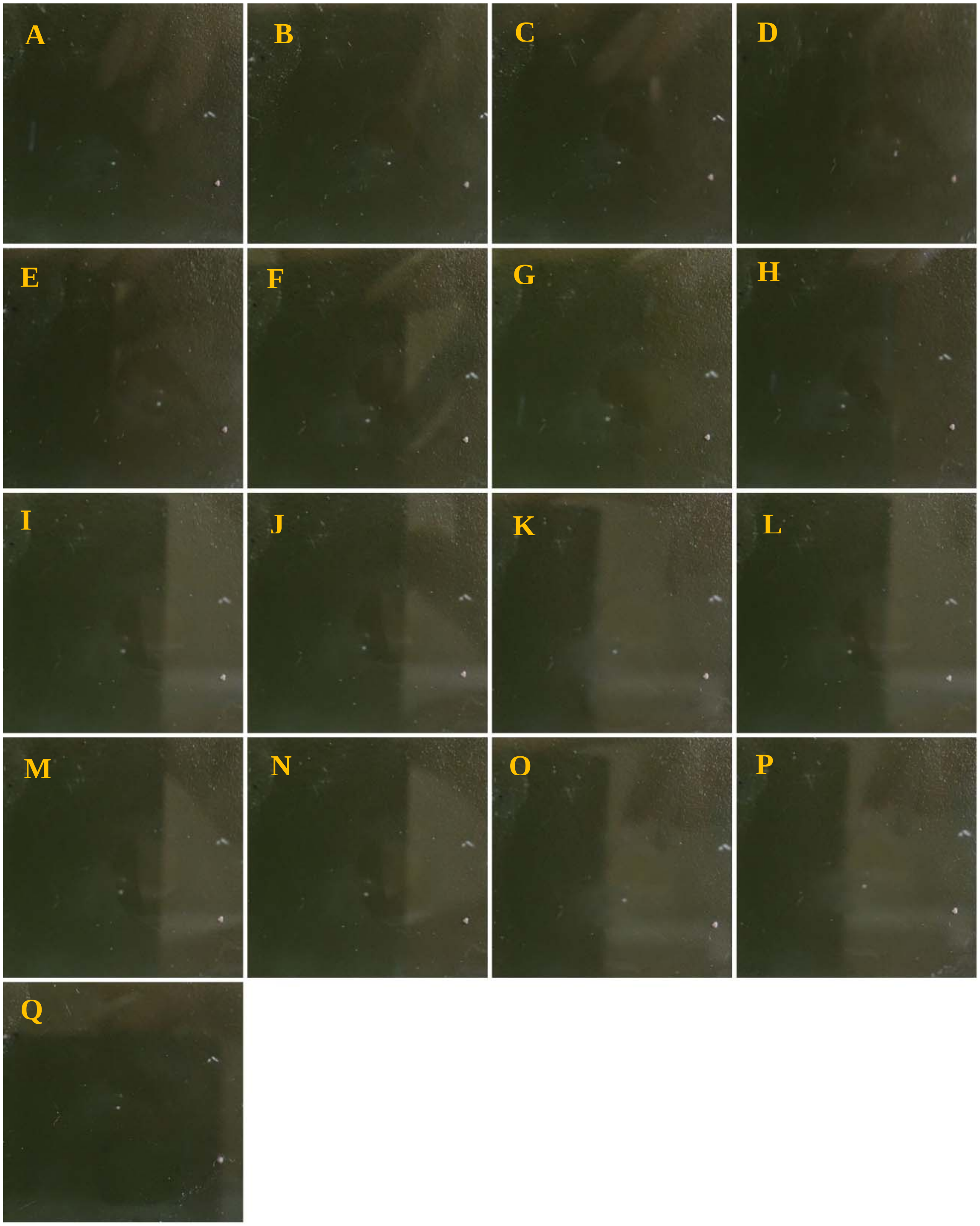


Fig. F2 — MES on the silicone oil AR20 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

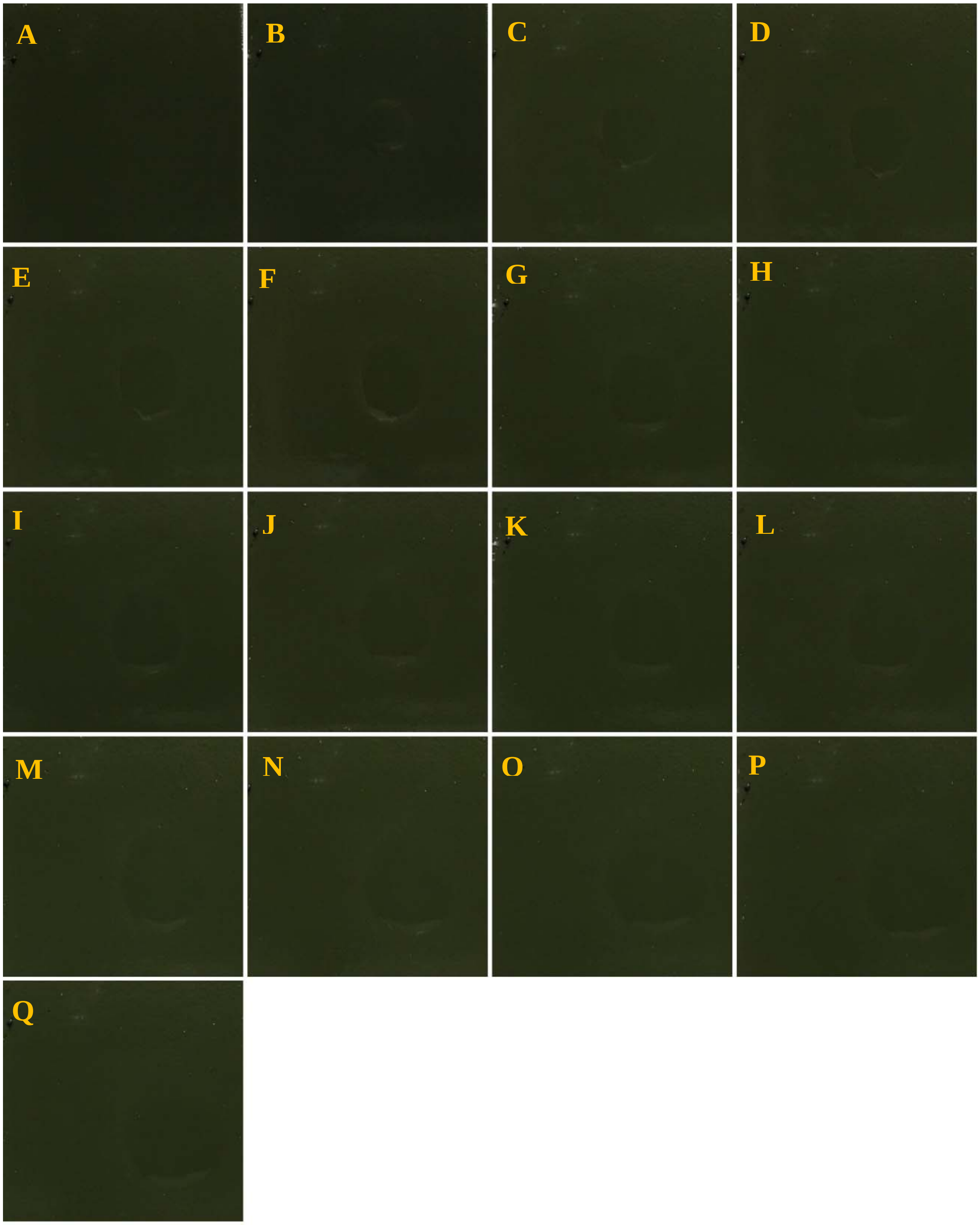
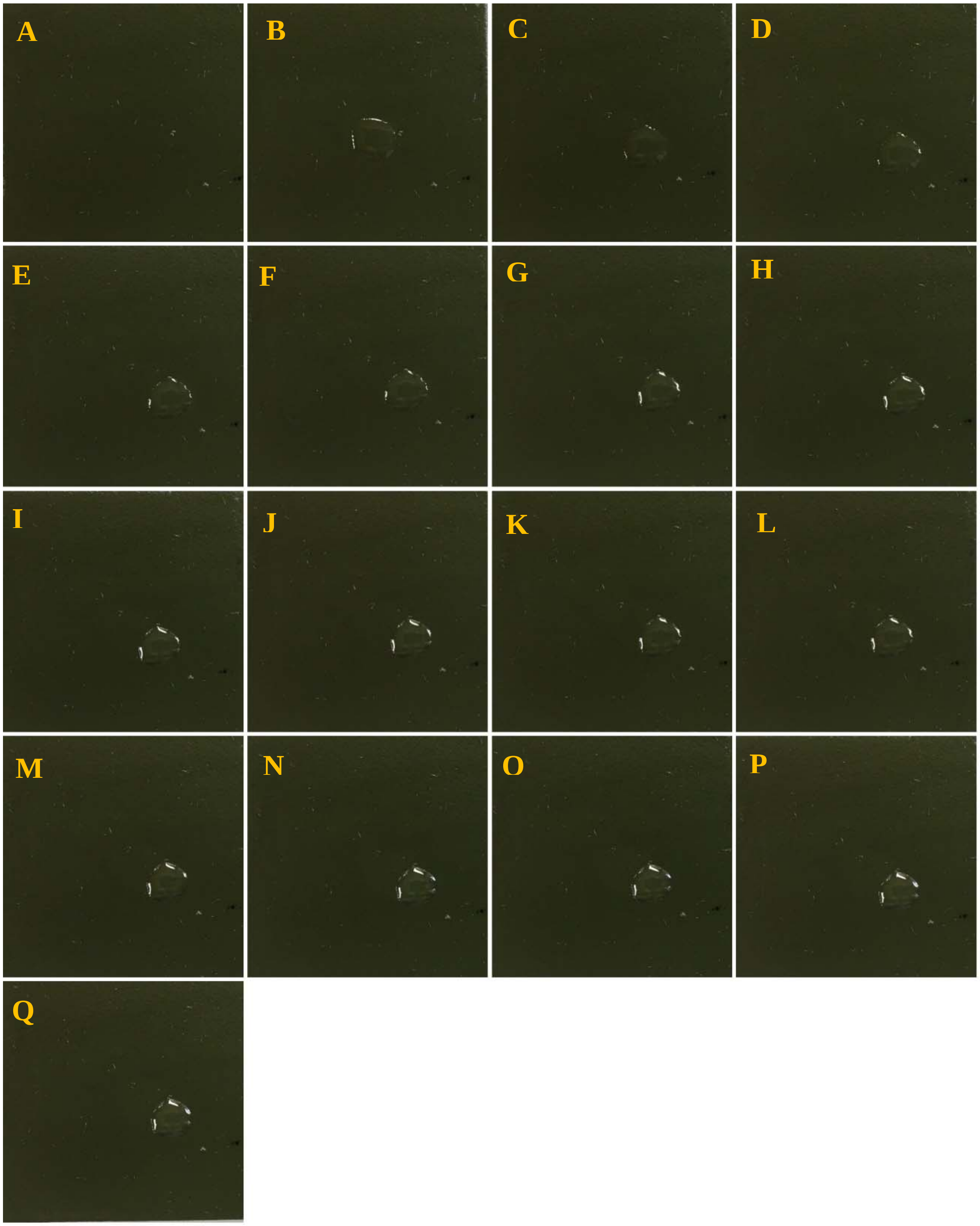


Fig. F3 — DMMP on the silicone oil AR20 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.



Appendix G

SILICONE OIL AR200 LUBRICATED PAINT IMAGES

Fig. G1 — DFP on the silicone oil AR200 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

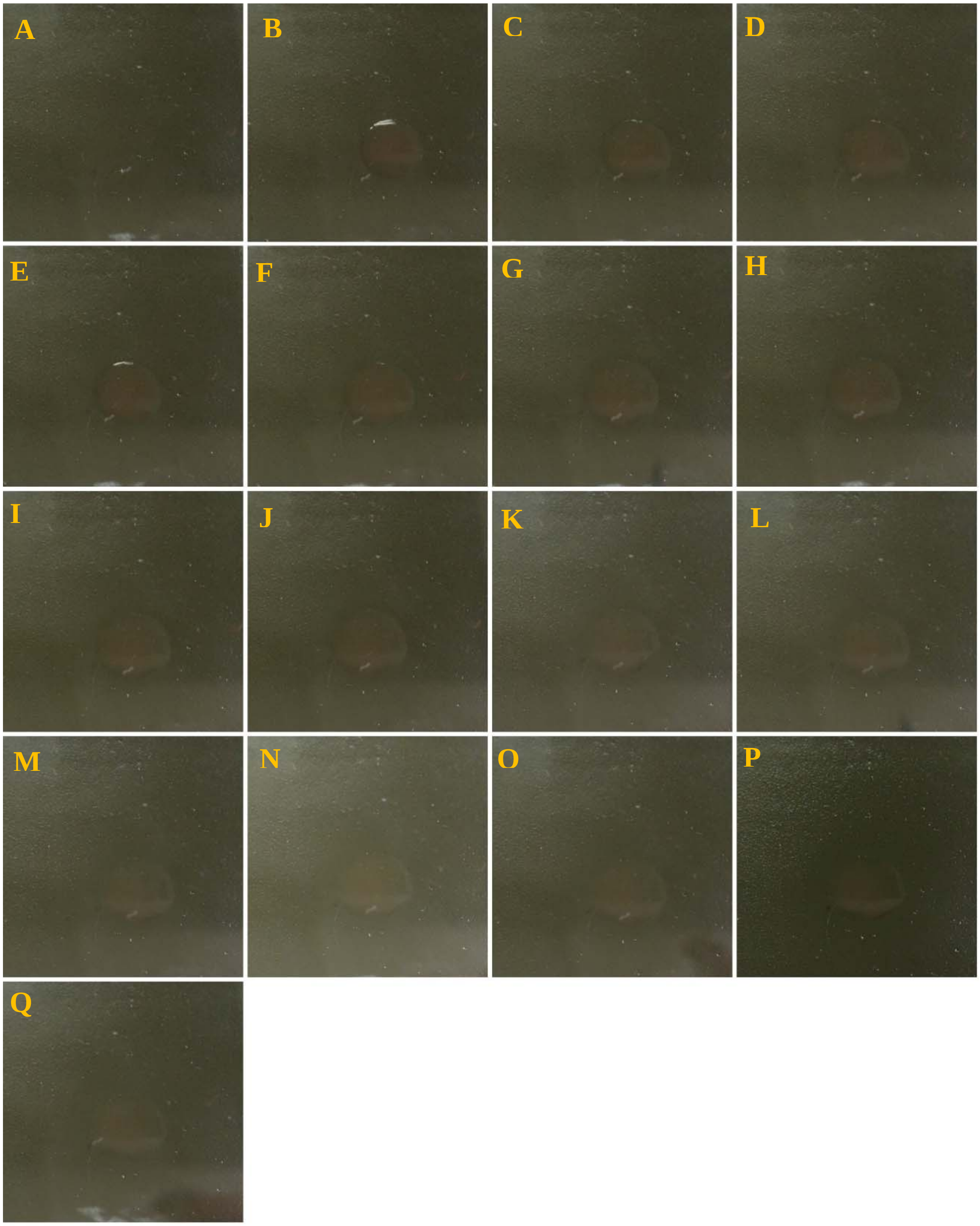


Fig. G2 — MES on the silicone oil AR200 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

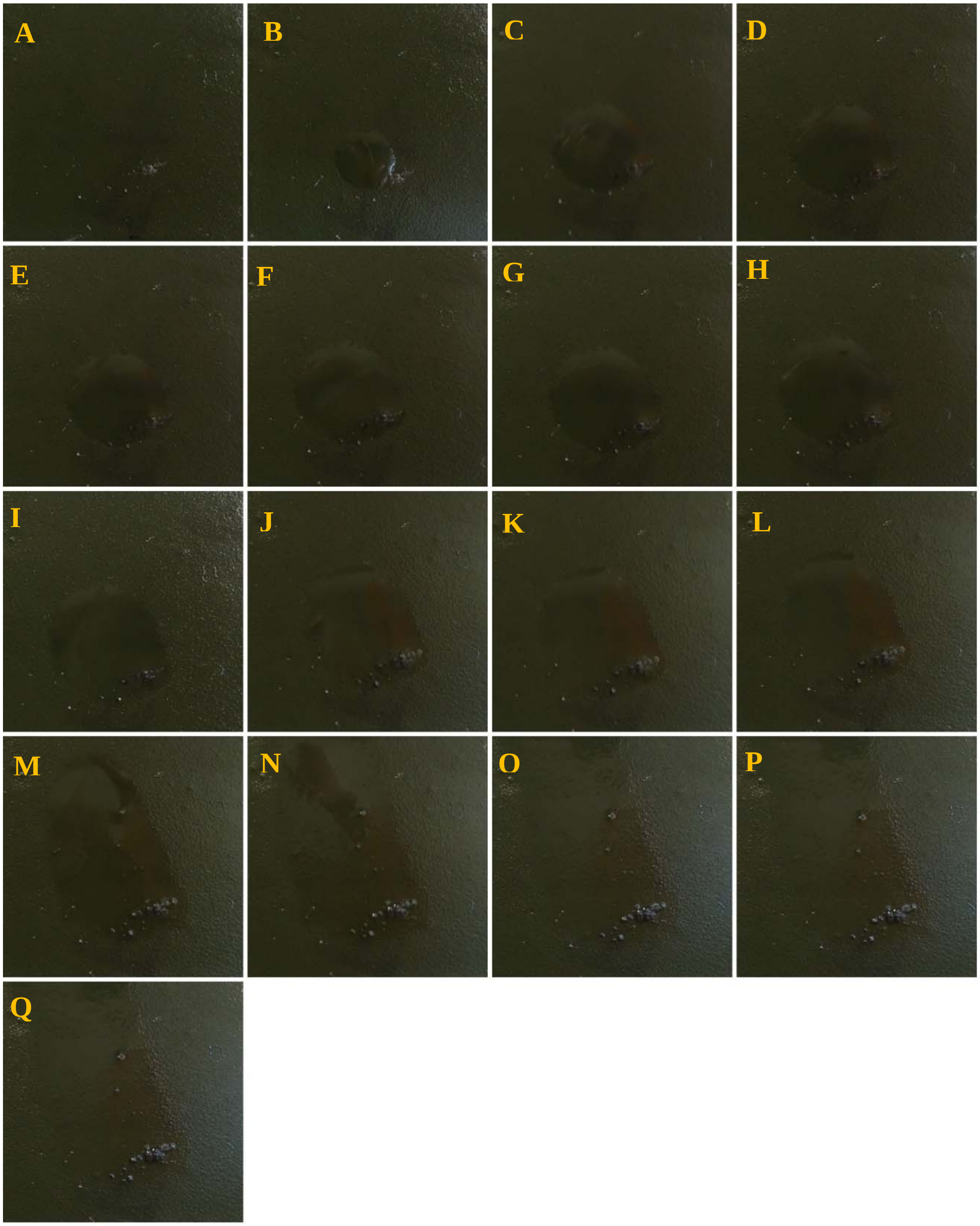
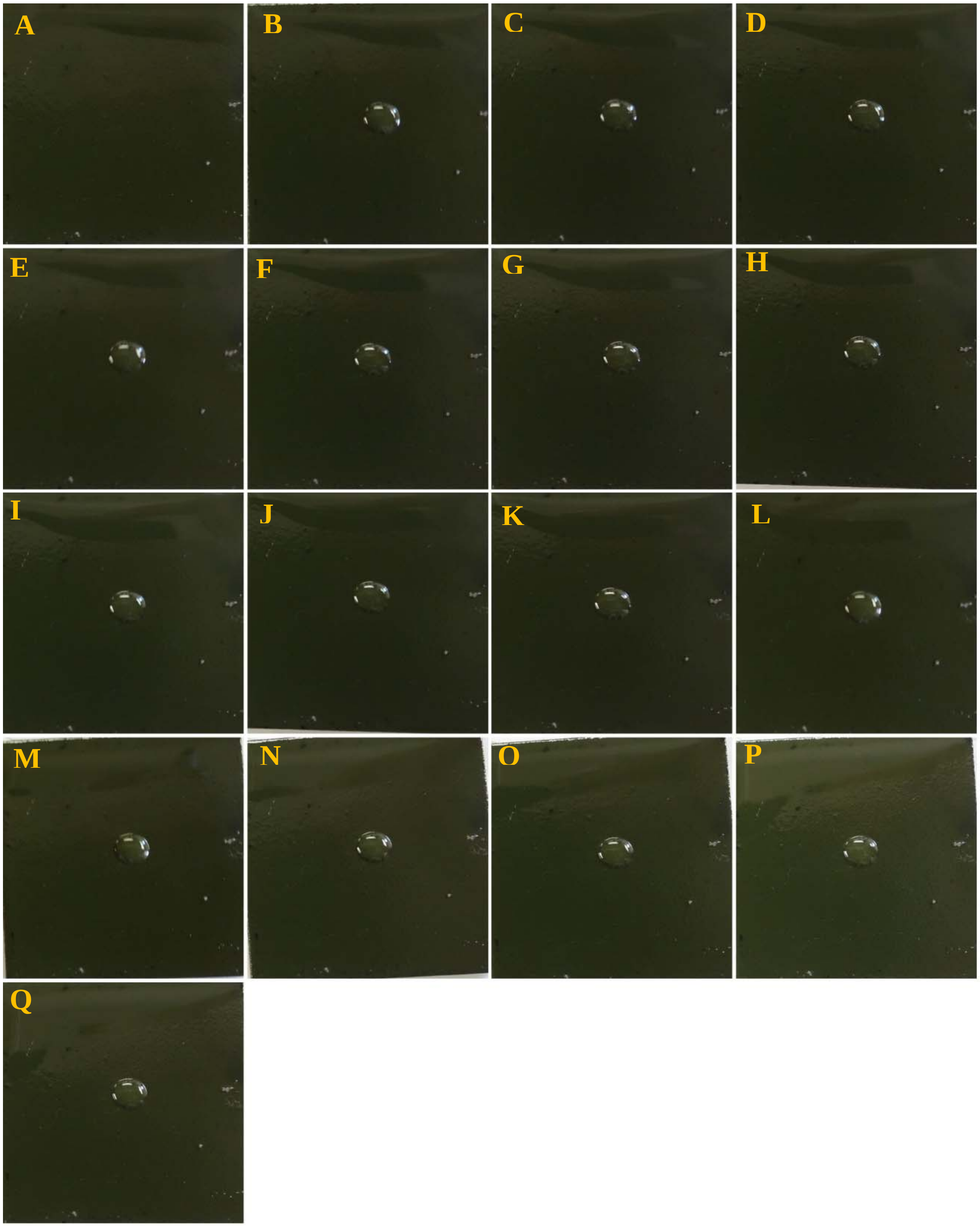


Fig. G3 — DMMP on the silicone oil AR200 lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.



Appendix H

DMPS 2X LUBRICATED PAINT IMAGES

Fig. H1 — DFP on the silicone oil DMPS 2X lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

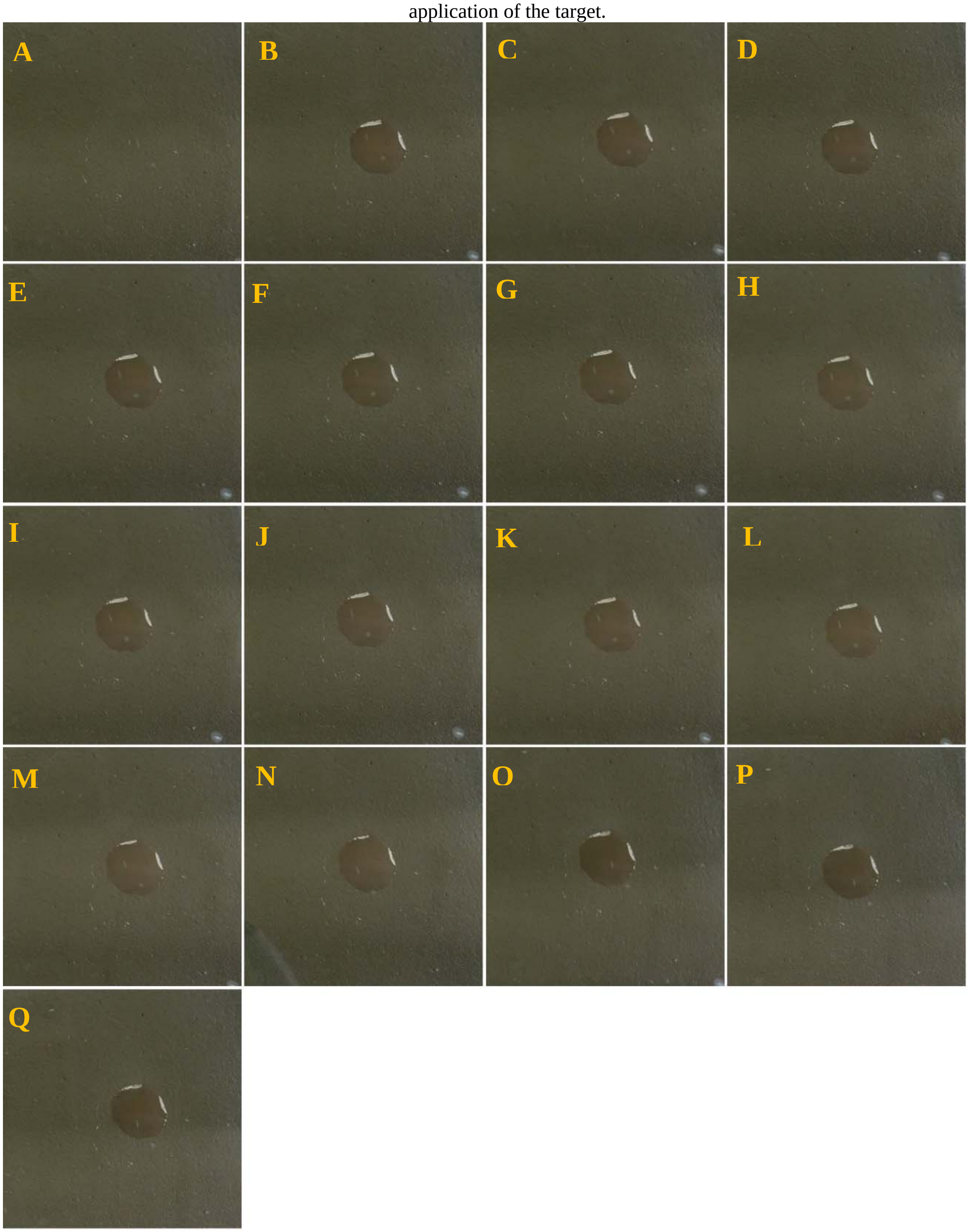


Fig. H2 — MES on the silicone oil DMPS 2X lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

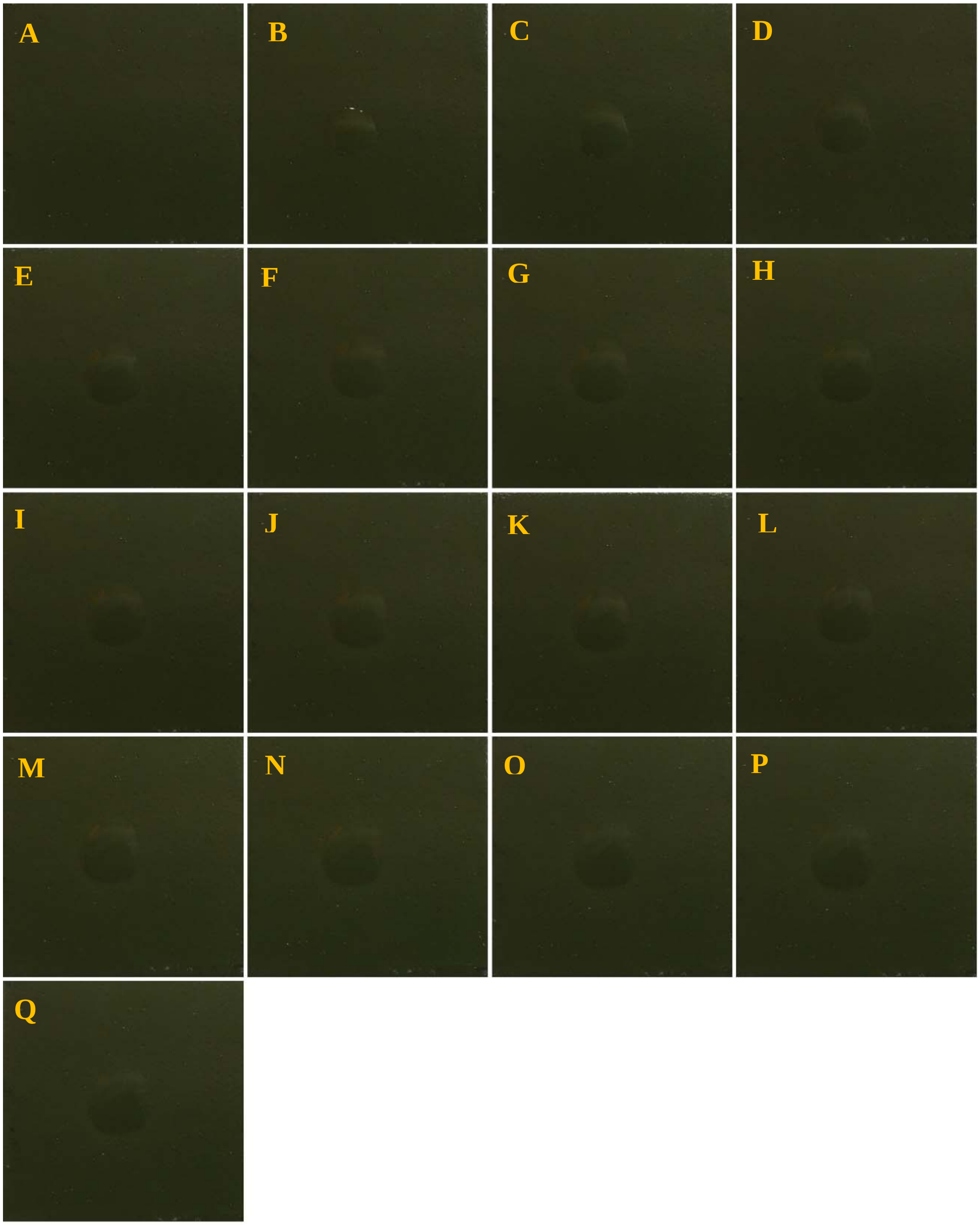
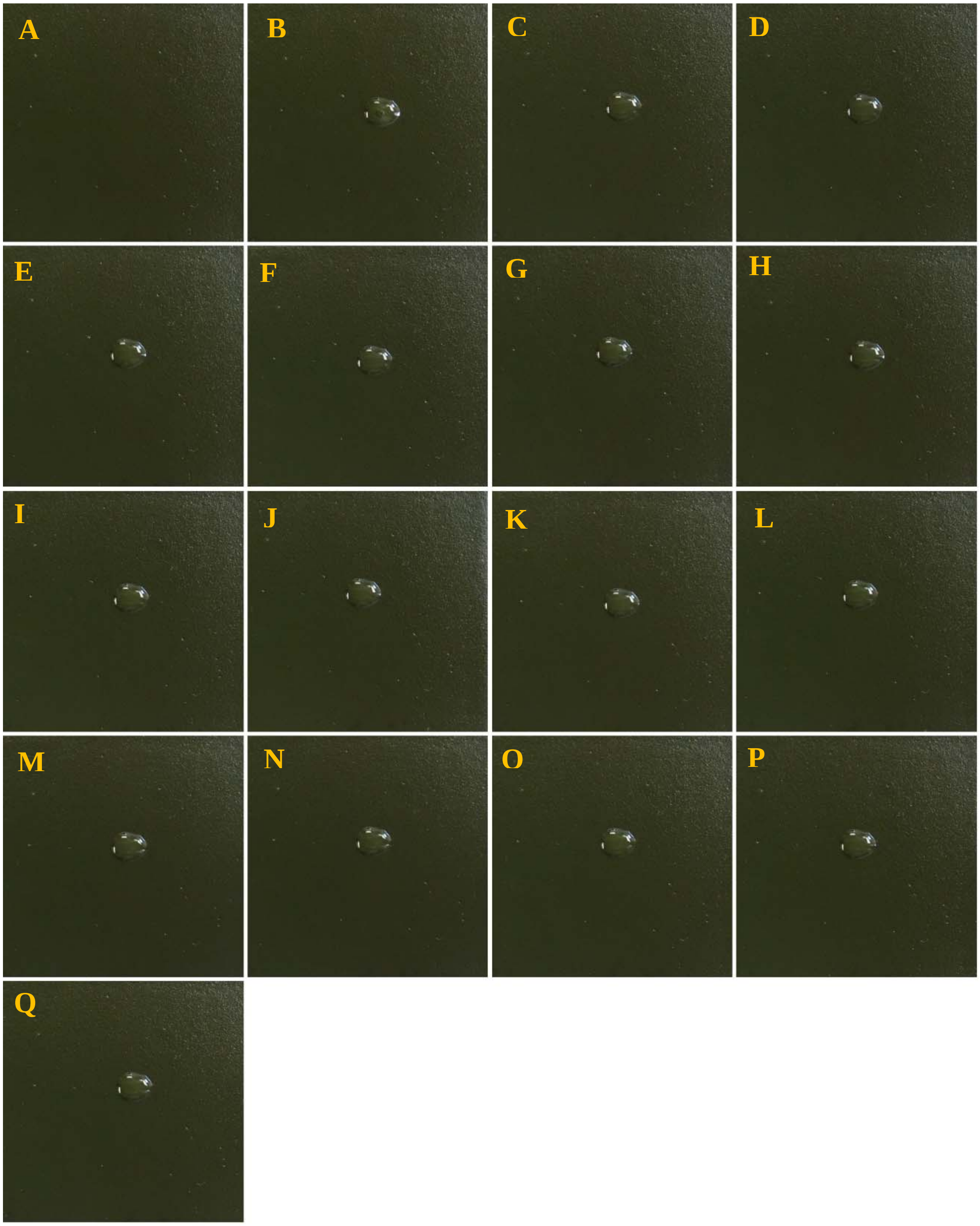


Fig. H3 — DMMP on the silicone oil DMPS 2X lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.



Appendix I

DMPS 5C LUBRICATED PAINT IMAGES

Fig. I1 — DFP on the silicone oil DMPS 5C lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

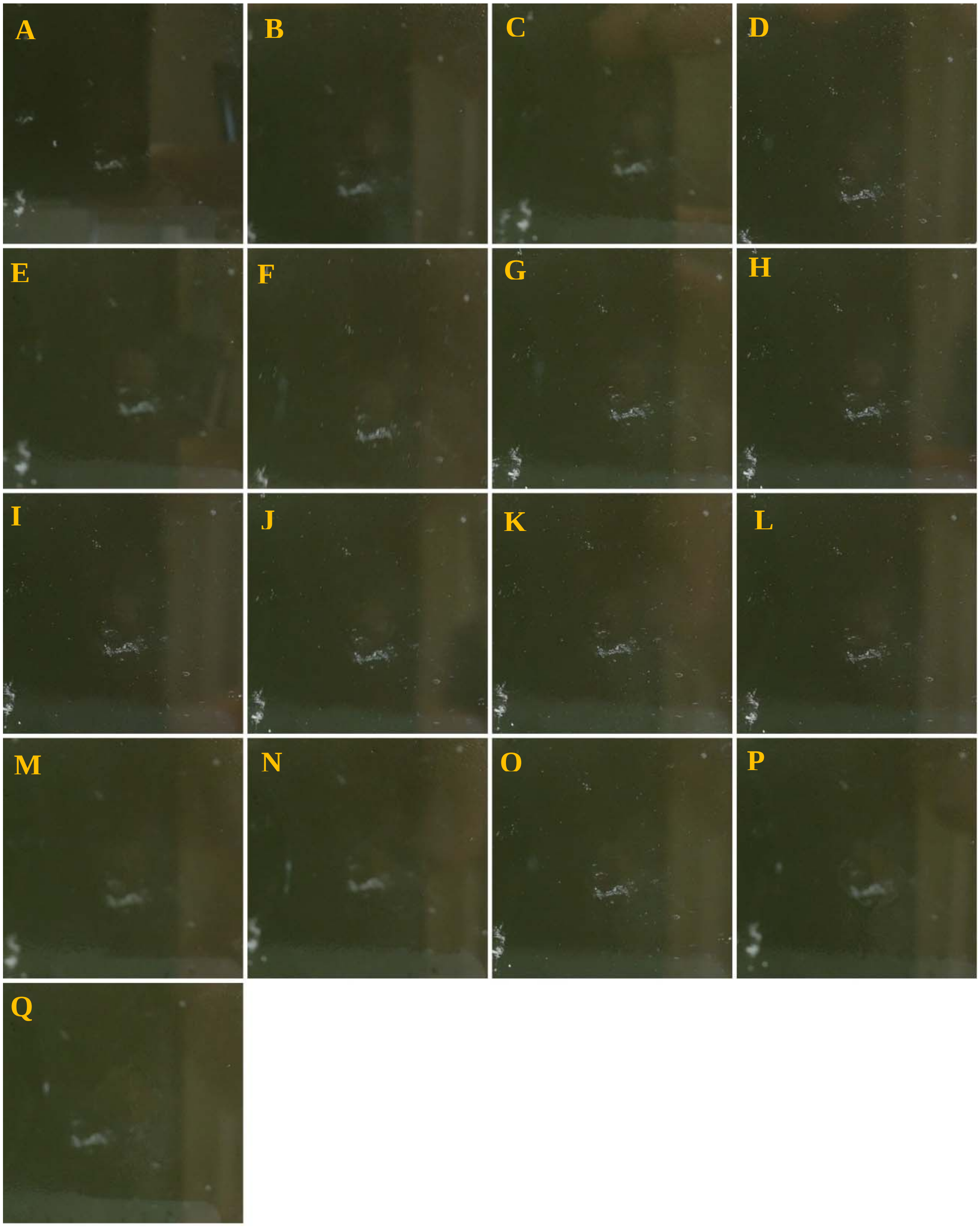


Fig. I2 — MES on the silicone oil DMPS 5C lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.

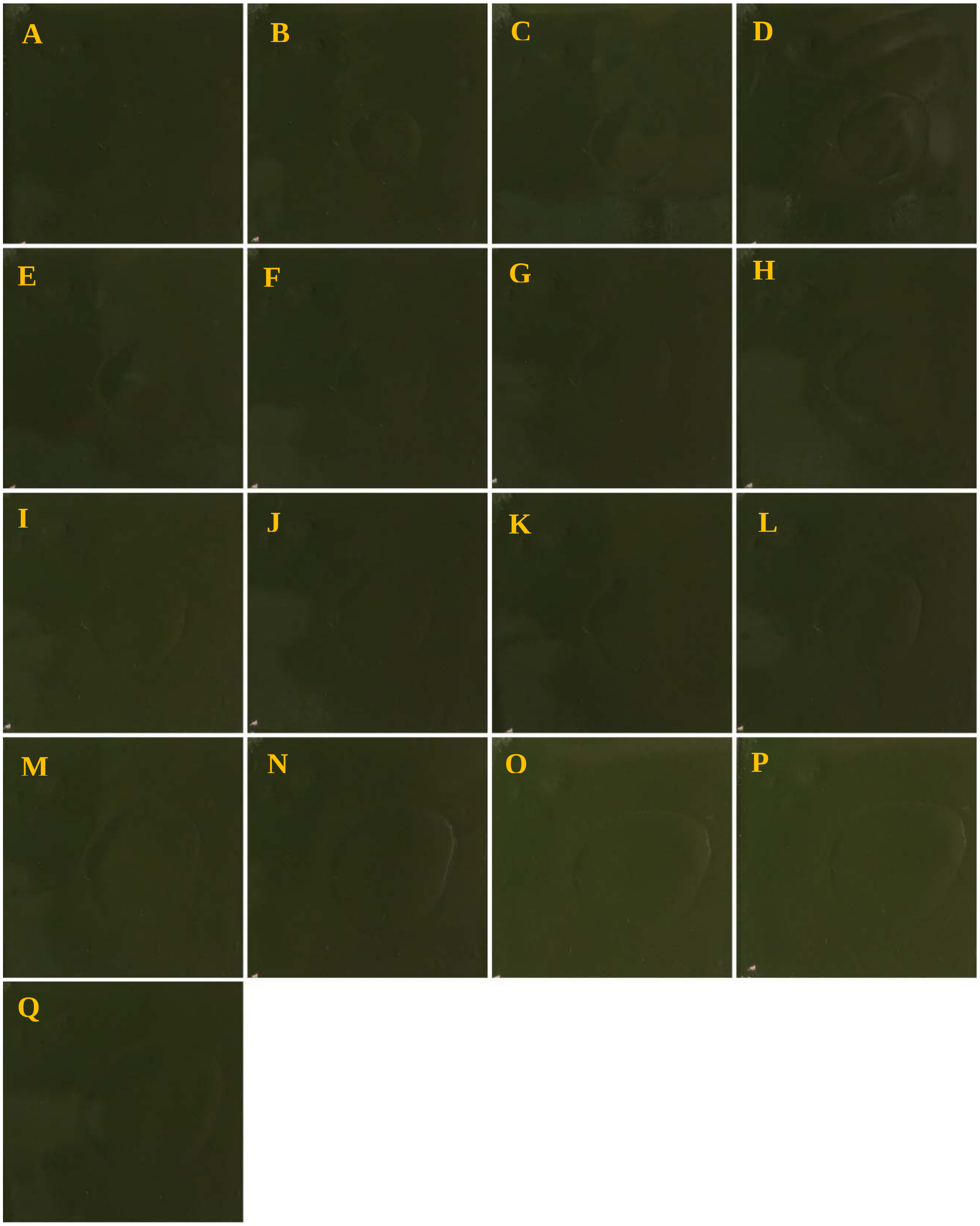


Fig. I3 — DMMP on the silicone oil DMPS 5C lubricated paint. Images of a coupon before application (A) and at 0 (B), 0.5 (C), 1 (D), 1.5 (E), 2 (F), 2.5 (G), 3 (H), 3.5 (I), 4 (J), 4.5 (K), 5 (L), 10 (M), 15 (N), 20 (O), 25 (P), and 30 (Q) min following application of the target.