



TEST AND EVALUATION STATUS REPORT 1 Updated

Characterization of Prototype Asphalt Shingle Rejuvenator, Invigosoy Roof ISR 0003

November 24, 2020

Report For: Iowa State University/CTRE
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Attn: Chris Williams, Ph.D.

Client: Iowa State University

Project No.: ISU 01-02-01

SAMPLE DATA / INFORMATION:

IDENTIFICATION	GRADE/TYPPE	DATE RECEIVED	SOURCE
Shingle Rejuvenator	Invigosoy Roof ISR 0003	8/27/20	Iowa State University
Aged Shingles	3 - Tab	8/30/2019	PRI Inventory

BACKGROUND: There are number of properties that must be met to characterize and qualify an asphalt shingle rejuvenator for potential commercialization. The properties are both technical (performance properties such as degree of penetration, shingle benefits, durability / life cycle, etc.), practical (methodology of application) and commercial (dilute ability & resultant stability; activate ingredient %, general method of application (spray, brush, other), shelf life, color change, etc.) See Diagram in Appendix.

Asphalt shingle and/or commercial BUR roofing rejuvenators have been used in the roofing for decades, with data going back to the 50's. Recently, the use of shingle rejuvenators has been reborn with the use of bio-oils in the pavement sector. The use of bio-oils brings a new technology with a multiplicity of options into the asphalt market.

The use of a shingle rejuvenator must provide benefit – increase the life cycle of the shingle and must retain critical performance properties of Fire Resistance, Hail Impact Resistance, Wind Resistance, Granule Retention, etc. Often a property may be improved at the expense of degrading another property, by example an application may change the color of the shingles, yet 'weather off' in time.

OBJECTIVE: Phase I: Initial screening evaluation and assessment of a prototype asphalt shingle rejuvenator, "Invigosoy Roof ISR 0003, to identify and quantify selected key performance properties to determine efficacy and potential commercial viability

CONCLUSIONS: Preliminary

See next page



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Summary Table:

Property	Best Results	Comments
Screening Properties		
Application Rate	200 ft ² / gal	at 4:1 dilution, improves upon aging
Flexibility	All 3 rates equal	retesting required; improvement noted at 100-105°F
Granule Adhesion Wet & Dry	200 ft ² / gal	Significantly better than 350 & 500 ft ² / gal – major benefit
Color Change	all	Darkens at 1 st , but then weathers off quickly
Next Step – Pahe 2: Comparative Evaluation of Aged Shingles With and Without Treatment (selected rate)		
Color Δ	200 ft ² / gal	Based on the collective results of Phase 1’s screening tests proceed to a modified Phase 2
Granule Adhesion, wet & dry		
Tear & Tensile Strength		Review means to improve: Penetration using additives. Rejuvenator stability and reduced ‘dripping’
UL 94 Fire Resistance		
Hail Impact Resistance / Pliability		
Mass Change of rejuvenator		
To Do: Final / Full Evaluations		
Color Change	Selected Application Rate and full scale testing	Based on these results, IP & commercialization would be recommended
Granule Adhesion		
Tear Strength		
Flexibility / Pliability		
Wind, Rain & Hail Impact Resistance		
Over Application Effects		

DISCUSSIONS:

Brief discussions are included after each separate property note in the Data / Results Tables.

DATA / RESULTS:

Table 2. Basic Physical Properties of Invigosoy Rejuvenator

PROPERTY		TEST METHOD	RESULTS	
			As Rec'd	Diluted *
SG	25°C	D70	0.9777	0.9968
Viscosity, cps	4°C	D4402 / D2196	164.4	18.4
	25°C		88.9	13.6
	60°C		49.8	9.8
Acid #		D664	2.22	0.51
Solubility _{Water}		Soxhlet	83.6	97.8

* Diluted: 4 parts water, one (1) part rejuvenator

Discussion: Diluted viscosity is low and may cause run off issues, whereby sprayed application runs off the roof (shingled roof have various slopes? Run off results in uneven application of the active ingredients. Non-water-soluble component (S) may be a plus.



Table 3A. Rejuvenator Penetration into Filter Paper as a Function of Application Rate & Time

RESULTS: Penetration Depth, mm & sheets, Based on Application Rate & Time							
Application Rates		1 hour after application		1 day after application		3 days after application	
		Depth, mm	Sheets	Depth, mm	Sheets	Depth, mm	Sheets
Invigosoy	200 sq. ft / gal	0.531	9	0.590	10	0.708	12
	350 sq ft / gal	0.472	8	0.531	9	0.590	10
	500 sq ft / gal	0.285	5	0.413	7	0.472	8

*Samples were conditioned at 120±5°F

DISCUSSION: We use filter paper as a quick screening to identify the potential penetration into a shingle. As expected, Higher coverage rates (less active ingredient) results in reduced penetration. Reduced penetration results typically in reduced benefits. The penetration improves with simulated exposure time after application.

Table 3B: Rejuvenator Penetration into Asphalt Coated Paper as a Function of Application Rate & Time

RESULTS: Penetration Depth, mm & sheets, Based on Application Rate & Time							
Application Rates		1 hour after application		1 day after application		3 days after application	
		Depth, mm	Sheets	Depth, mm	Sheets	Depth, mm	Sheets
Invigosoy	200 sq. ft / gal	0.889	2	1.3	3	1.3	3
	350 sq ft / gal	0.889	2	0.889	2	0.889	2
	500 sq ft / gal	.508	1	0.508	1	0.508	1

DISCUSSION: Asphalt coated 'rigid' paper is a surrogate for the shingles, the penetration results followed those of the Filter Paper. For reference, the single layer thickness on typical architectural / dimensional shingles is approximately 2 - 4 mm w. granules and 1-3 mm for the asphalt filled coating only. Penetration into the full depth of the asphalt coating is preferred. The coated paper has a thin layer of coating (higher SP) on either side of a rigid paper, it does not impregnate with asphalt. Penetrations did not improve with time at the 350 and 500 ft ²/ gal coverages.

Table 3C: Rejuvenator Penetration Into # 30 Asphalt Saturated Felt as a Function of Application Rate & Time

RESULTS: Penetration Depth, mm & sheets, Based on Application Rate & Time							
Application Rates		1 hour after application		1 day after application		3 days after application	
		Depth, mm	Sheets	Depth, mm	Sheets	Depth, mm	Sheets
Invigosoy	200 sq. ft / gal	1.65	1	1.65	1	1.65	1
	350 sq ft / gal	0	0	0	0	0	0
	500 sq ft / gal	0	0	0	0	0	0

DISCUSSION: # 30 Asphalt Saturated Felt, is an ASTM defined product which is an organic felt (made recycled cellulose fiber via a wet process (not woven) asphalt impregnated though the entire thickness. This screening evaluation will assess the penetration through a material that more closely replicates an asphalt shingle employing filled coating. More to follow upon completion.


Table 4. Shingle Flexibility/Cracking vs. Application Rate via Mandrel Bend Test

Flexibility Test * on Aged Shingles at Three Application Rates				
Temperature, °F	Control	200 sq ft/gal	350 sq ft/gal	500 sq ft/gal
74	3/3 Fail	3/3 Fail	3/3 Fail	3/3 Fail
85	3/3 Fail	3/3 Fail	3/3 Fail	3/3 Fail
95	3/3 Fail	3/3 Fail	3/3 Fail	3/3 Fail
99	3/3 Fail	3/3 Fail	3/3 Fail	3/3 Fail
104	3/3 Fail	3/3 Pass	3/3 Pass	3/3 Pass
110	3/3 Fail			
115	3/3 Fail			
118	3/3 Pass			

* 1x6 inch aged shingle pieces were treated with rejuvenator and cured for 3 days at 140°F. Samples were then conditioned at different temperatures for 1 hour and were bent 180° around a mandrel in 2 seconds. Mandrel size 1 inch.

DISCUSSION:

These results suggest an improved flexibility / pliability of a treated aged shingle is minimal. A more flexible / pliable shingle Improves Hail Impact Resistance and improves the shingles response to weather induced expansion and contraction.

Table 5A. Granule Adhesion (Dry) of Treated Aged Shingles

APPLICATION RATE		Granule Loss, g (%) by Shingle Tab					
		Tab 1		Tab 2		Tab 3	
		200 sq ft/gal		350 sq ft/gal		500 sq ft/gal	
		Untreated	Treated	Untreated	Treated	Untreated	Treated
Loss	g	-0.41	-0.12	-0.40	-0.16	-0.47	-0.14 **
	%	-0.95	-0.24	-0.79	-0.35**	-1.07	-0.29 **

* Samples, 2" x 9" were conditioned at 140±5°F for 1 week, tested at 25°C
 ** data suspect, retesting

DISCUSSION: %'s of improved granules adhesion: 71%; 63% and 78% respectively the 500 ft²/gal is being retested. Improved granule adhesion is an extremely important property and benefit. Granules are instrumental in reducing the aging of the shingles via shielding the filled coating from, UV light – oxidative aging, and water – erosion due to formation of water-soluble compounds due to oxidative aging. The granules are primary for fires resistance classifications and favorably provide weight to the shingle improving wind resistance and provide astatic appeal. We tested granule adhesion after 1 week at 140°F (typical roof top temperatures) to better simulate real world conditions. However, this does not reflect overall durability (the life cycle of the rejuvenator.0).



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Table 5B. Granule Adhesion (Wet) of Treated Aged Shingles

APPLICATION RATE		Granule Loss, g (%) by Shingle Tab					
		Tab 1		Tab 2		Tab 3	
		200 sq ft/gal		350 sq ft/gal		500 sq ft/gal	
		Untreated	Treated	Untreated	Treated	Untreated	Treated
Loss	g	-1.45	-0.88	-1.09	-0.95	-0.96	-0.99
	%	-3.38	-1.90	-2.40	-2.14	-2.14	-2.26
Samples, 2" x 9" were conditioned at 140±5°F for 1 week, tested at 25°C after 1 hr. water soak at 25C							

DISCUSSION: provides information on the impact of granule adhesion after water conditioning a more sever condition. % improvements were 148%; 26% and 0% respectively. 500 ft²/gal is being retested.

Table 6A. CIELAB Color and Change Comparison of Treated vs. Untreated After 1,000 hrs. of Accelerated Weathering

Shingle Treatment	Color Change by Colorimeter							
Blend	Hours	L*	a*	b*	Δ L*	Δ a*	Δ b*	Δ E*
Control (0 Treatment)	0	50.77	-1.12	0.18	0	0	0	0.00
	250	50.85	-1.16	0.12	0.08	-0.04	-0.06	0.11
	500	50.26	-1.12	0.26	-0.51	0	0.08	0.52
	750	48.54	-1.17	1.01	-2.23	-0.05	0.83	2.38
	1000	48.85	-1.18	0.76	-1.92	-0.06	0.58	2.01
200 sq ft /gal	0	46.14	-1.21	2.52	-4.63	-0.09	2.34	5.19
	250	49.41	-0.62	2.93	-1.36	0.50	2.75	3.11
	500	52.36	-0.39	1.77	1.59	0.73	1.59	2.36
	750	51.90	-0.37	1.60	1.13	0.75	1.42	1.96
	1000	52.28	-0.48	1.78	1.51	0.64	1.60	2.29
350 sq ft /gal	0	46.41	-1.34	2.27	-4.36	-0.22	2.09	4.84
	250	50.63	-0.84	3.21	-0.14	0.28	3.03	3.05
	500	52.66	-0.76	1.45	1.89	0.36	1.27	2.31
	750	55.20	-0.56	1.55	4.43	0.56	1.37	4.67
	1000	53.85	-0.75	1.78	3.08	0.37	1.60	3.49
500 sq ft /gal	0	49.04	-1.43	2.75	-1.73	-0.31	2.57	3.11
	250	48.78	-0.81	2.27	-1.99	0.31	2.09	2.90
	500	52.64	-0.54	1.51	1.87	0.58	1.33	2.37
	750	54.04	-0.67	1.70	3.27	0.45	1.52	3.63
	1000	54.00	-1.18	1.13	3.23	-0.06	0.95	3.37



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Table 6B. Explanation of Color Values

VALUE	EXPLANATION	POSITIVE VALUE	NEGATIVE VALUE
ΔL^*	Difference in lightness/darkness value	Lighter	Darker
Δa^*	Difference on red/green axis	Redder	Greener
Δb^*	Difference on yellow/blue axis	Yellower	Bluer
ΔE	Total color difference value	Nearer to zero is closer to the color of the control at 0 hours	

Discussion: The Colorimeter provides color shades changes due to exposure in a QUV weatherometer (used predominately for color change). The data exhibits and defines the color change and the rate of change

Table 7. Comparative Visual Representation of Shade Change Over QUV Exposure Time

QUV Exposure Hours	Rejuvenator Application Rates			
	Control		Invigosoy Roof ISR 0003	
	No Treatment	200 Sq Ft gal	350 Sq Ft / gal.	500 Sq Ft/ gal
0				
250				
500				
750				



1000

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DISCUSSION:

The Shades provide an additional method of indicating. Viewing the initial, 0 hrs. of exposure the application of the rejuvenator darkened the shingles in a proportionate to the % of active ingredient (not water) applied. Exposure in the QUV weatherometer, exhibits a shade change (becomes lighter – 'weathers off' as a function of exposure time.

"The QUV accelerated weatherometer reproduces the damage caused by sunlight, rain and dew. Its' primary focus is on color fastness. In a few weeks or months, the QUV UV weatherometer may replicate the damage (mostly color change) that occurs over months or years outdoors. The QUV does provide a degree of related outdoor aging. We use the QUV as a reduced cost screening tool for color fastness (rejuvenators tend to darken the shingle, which may or may not be an aesthetic issue),

To better simulate outdoor weathering in Phase 2 and subsequent Phases we will use the Xenon Arc accelerated weatherometer. The Xenon Arc weatherometer exposes materials to alternating cycles of Sunlight and/or Darkness and rain at approximated 50°F, with the specimens having a black body temperature of approximately 140°F. The Xenon Arc better simulates outdoor weathering in a very accelerated fashion. The Xenon Arc Weatherometer (XOM) is considered the industry standard for roofing asphalt coatings and is used by nearly all roofing product manufacturers. Its limitations are the small sample sizes (limits the type of testing that may subsequently be performed on the specimens). The XOM does an excellent job inducing accelerated oxidative aging and thermal shock which causes asphalt erosion and cracking.

PRI has developed the APWS, Asphalt Pavement Weathering System, which is also suitable for roofing materials. The APWS is a larger version of the XOM, providing up to 20 ft² of exposure area, where full shingles and mini roofs have been evaluated. The APWS has the same flexibility w.r.t. cycle controls with enhanced sensing and recording to include rain collection for possible leachate / environmental testing

OVERALL DISCUSSION:

After these screening evaluations the prototype rejuvenator exhibited, for the most part, competitively superior benefits when compared to the rejuvenators PRI has previously tested and evaluated. The key benefits were granule adhesion, minimalist color change, ease of application, and the collective overall benefits based on active component percentage.

The screening properties are sufficient to move into Phase 2 modified to address further work on penetration and possible dripping. In Phase 2 work will be performed on aged shingles will be treated, evaluated and assessed. These shingles are in good shape and represent shingles exposed for approximately 13-15 years in Clearwater, FL.

RECOMMENDATIONS:

Phase 2 will complete Items 3a-3i from Phase 1 on the aged shingles expand the property evaluations from screening to 'full scale'. Focus includes:

- A. improving hail impact resistance (penetration),
- B. review of fire resistance (Spread of flame, Intermittent Spread of Flame and Burning Brand)
- C. improvement is pliability / flexibility (wind uplift resistance)
- D. physical properties of Tear & Tensile
- E. Select properties to evaluate the overall durability of the treatment



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Iowa State:

- A. Improve stability of diluted product
- B. Improve / thicken viscosity to reduce potential drip issues
- C. Investigate concepts of using the penetrating aid (acts as a carrier for the rejuvenator)
- D. Review the concept of the ability to dilute with water (emulsion) this may cause application issues (contractors further dilute) which subsequently reduces benefits
- E. Initiate IP, if not already started.

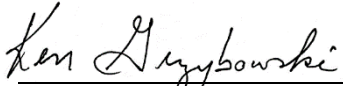
Tested by:



Fischer Sutherland, Binder Lab Technician

Date: November 24, 2020

Reviewed by:



K.F. Grzybowski, President

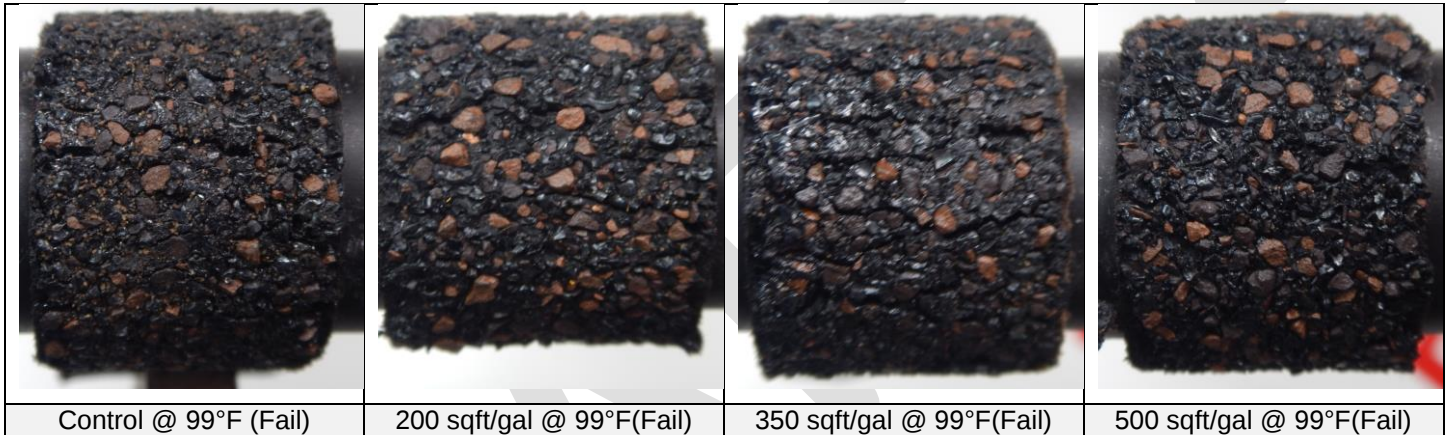
Date: November 24, 2020



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APPENDIX: Photos: Test Specimens Before & After Treatment

FLEXIBILITY / PLIABILITY: 1" diameter mandrel

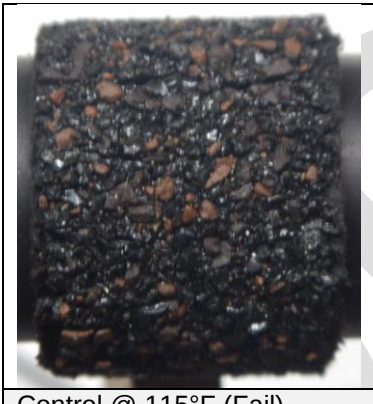




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Control @ 110°F (Fail)

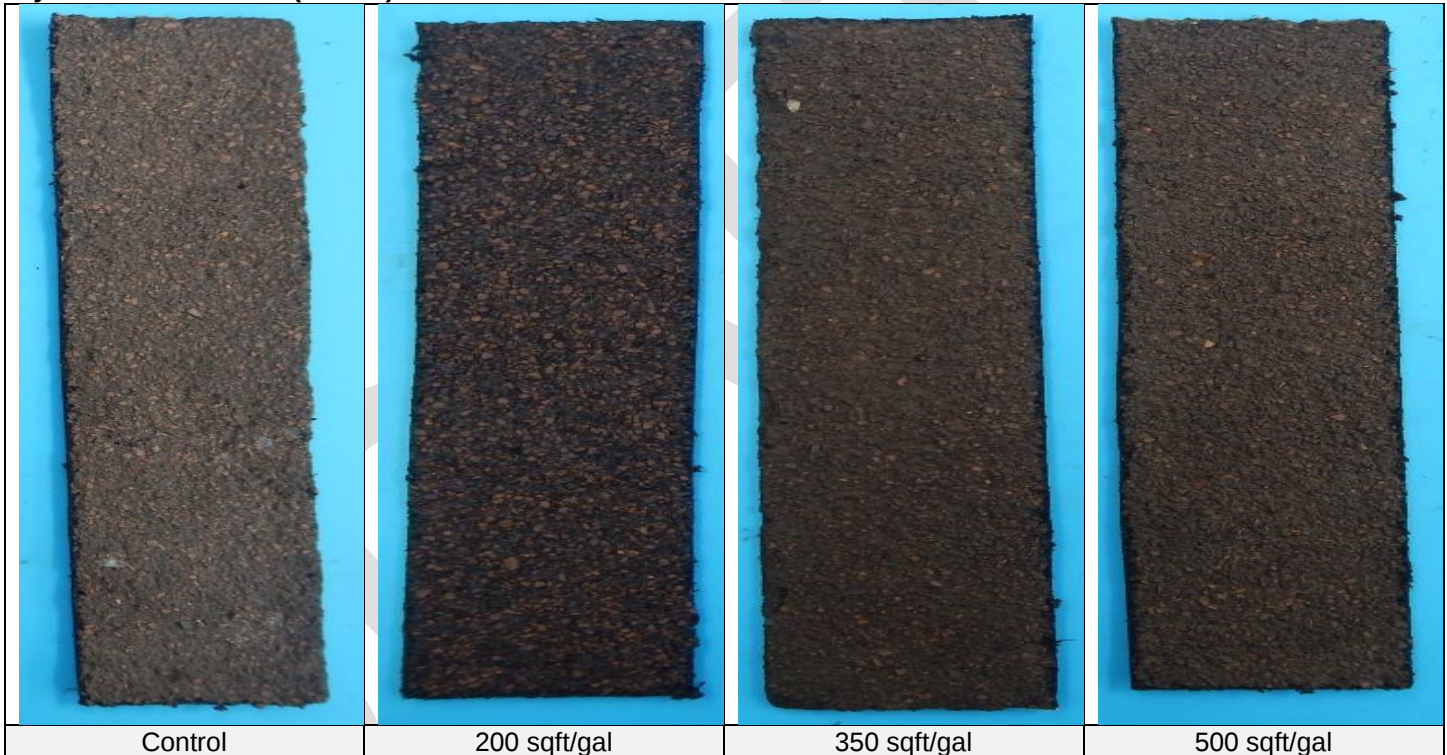


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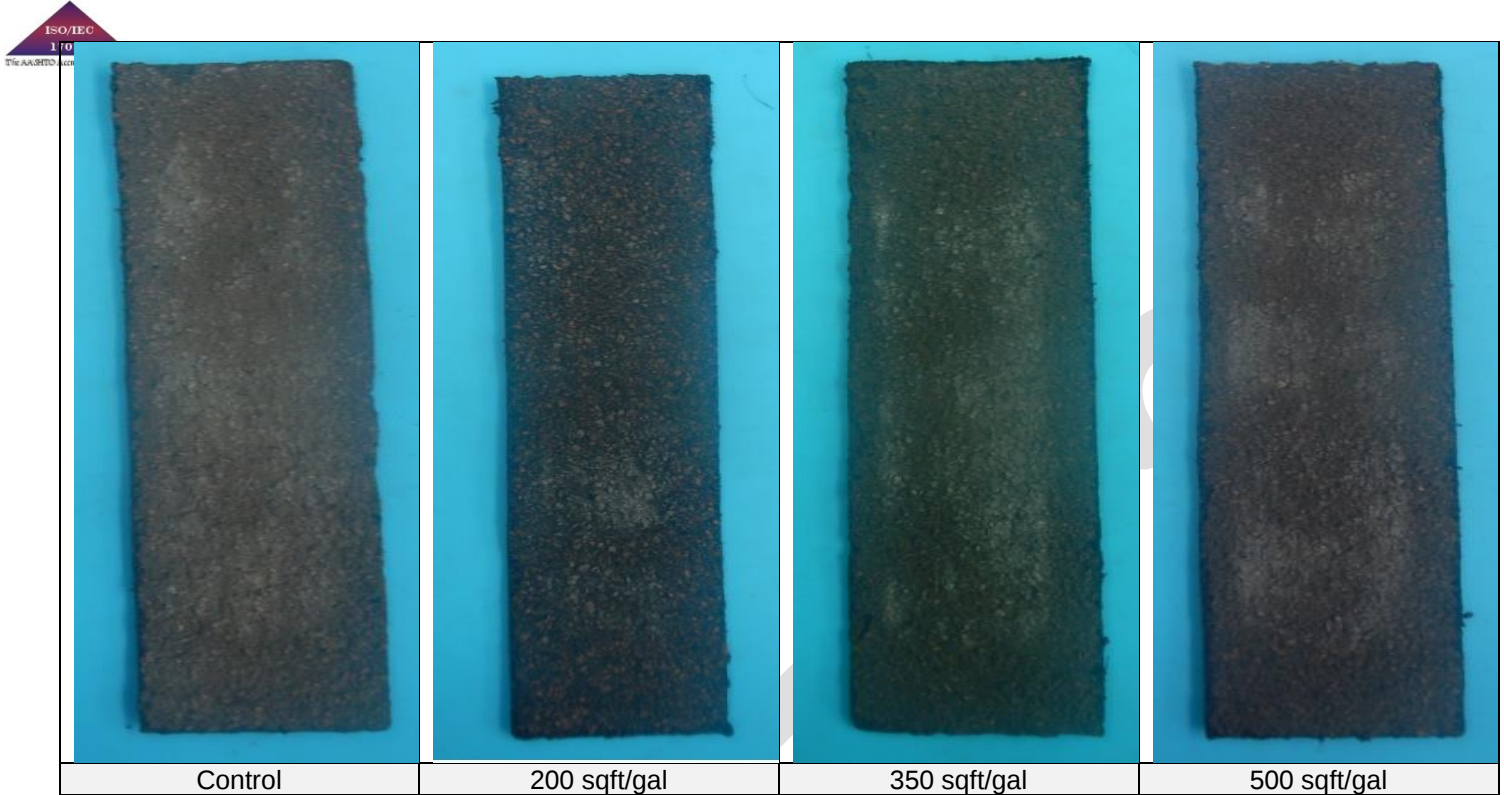


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Dry Granule Adhesion (Before)

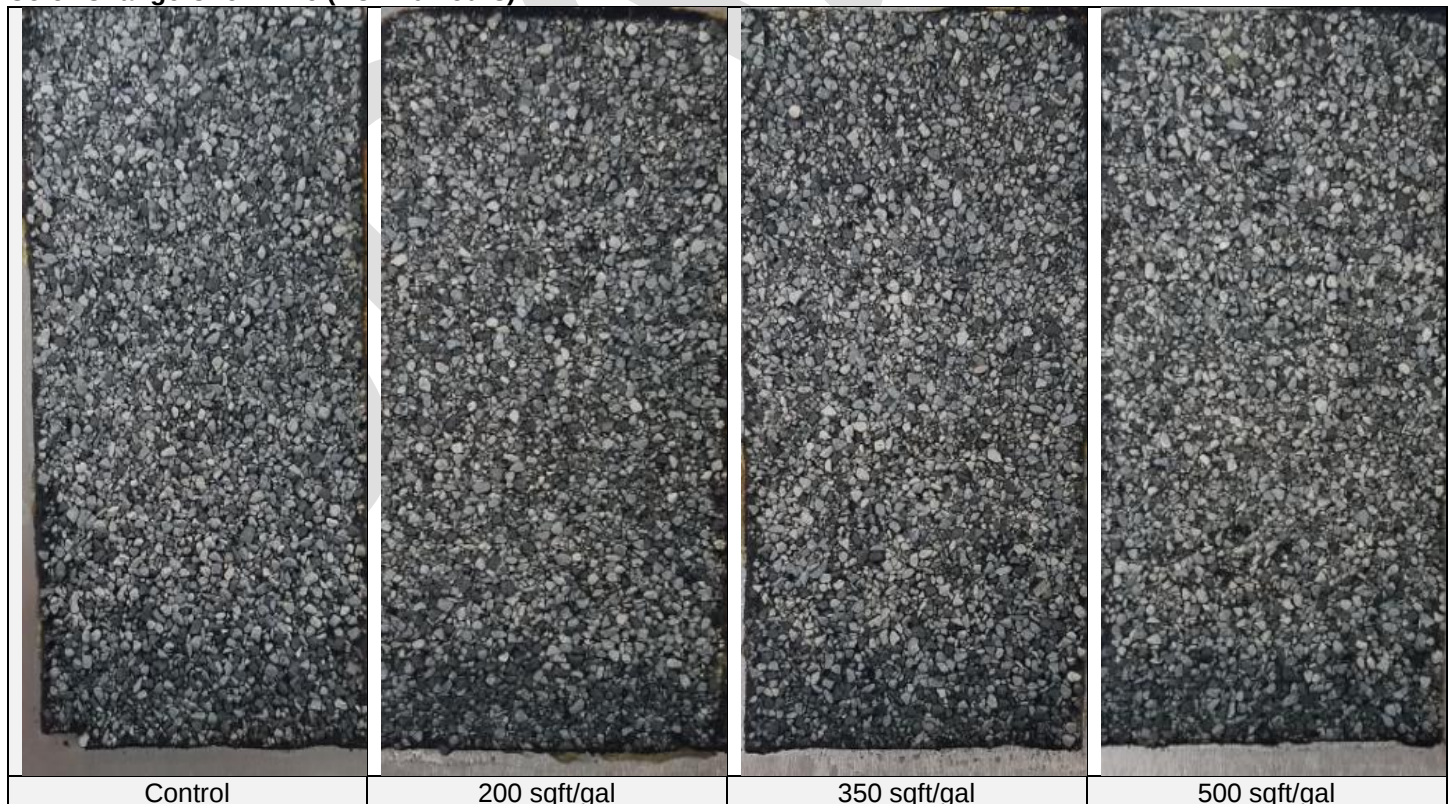


Dry Granule Adhesion (After 50 Cycles)



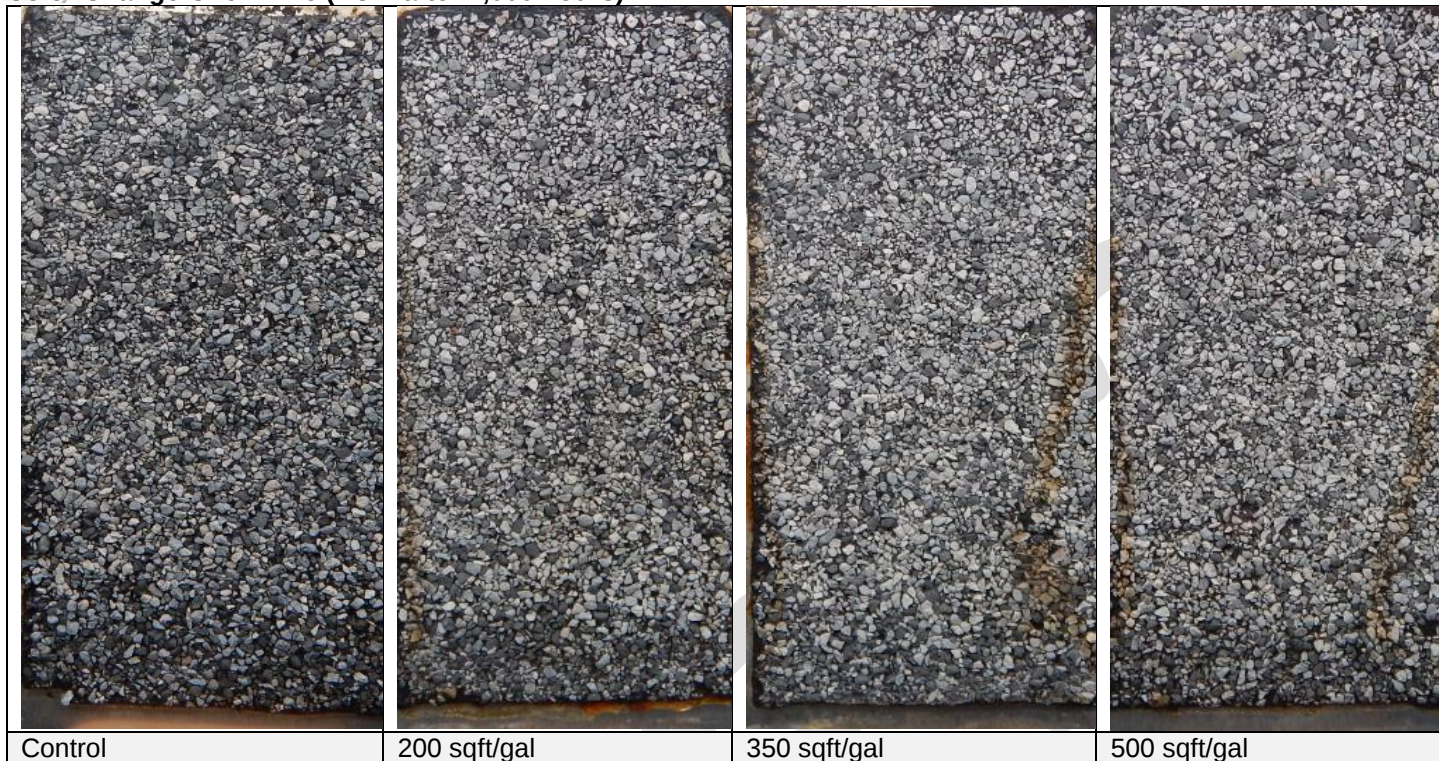
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Color Change Over Time (XOM 0 hours)





Color Change Over Time (XOM after 1,000 hours)

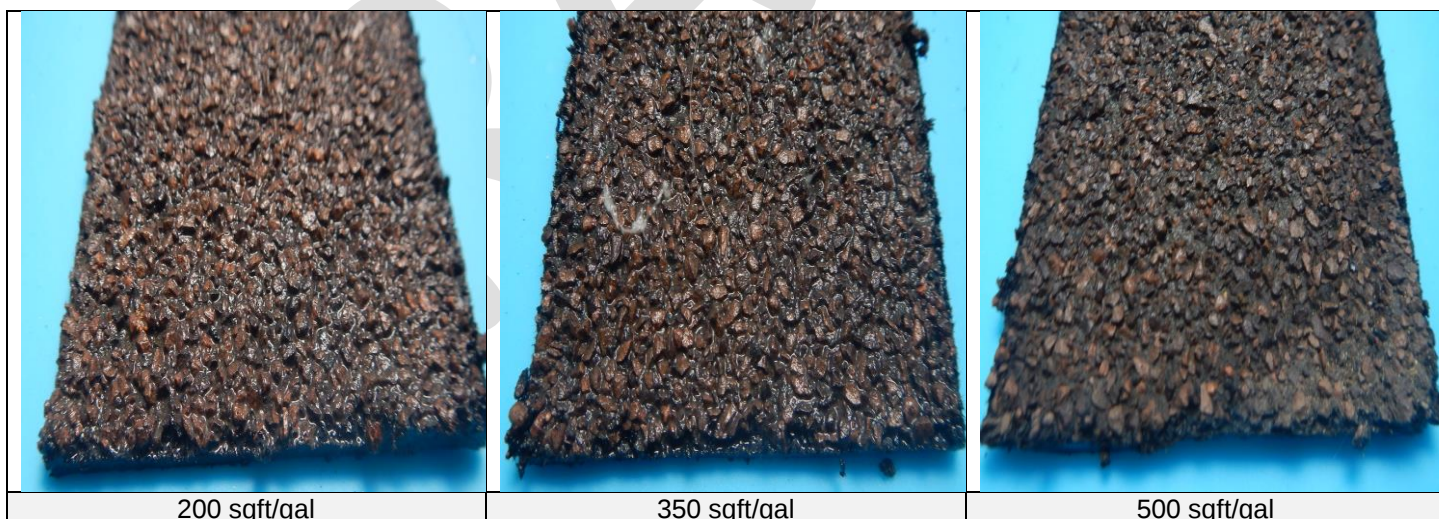


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Photos right after application at different rates (for Run-off assessment)





SHINGLE REJUVENATOR REQUIREMENT GUIDELINE

Supportive Literature

- SDS - wt./gal., flash point, etc.
- TDS (from Technical Data)
- Marketing (TBD)

Technical Data

- Degree of penetration/depth
- At various application rates
- With candidate formulas
- Impact on coating
- Physical Properties: SP, Pen, Duct., SARA Fractions
- Carbonyl & Sulfoxides
- Effects on Aging
- Granule Adhesive Interface
- Oxidation/weathering rates (H₂O Solubility)

Potential Environmental Impacts (Over Spray, Dripping, Spillage)

- Vegetation/Landscapes
- Leachate
- PAH's
- VOC's

Shingle Rejuvenator Commercialization

Guideline Requirements

Formula Development

- Rejuvenation Capability - Cargill Veggie Based Oil
- Penetration - tall oil derivative (carrier for rejuvenating oil)

Deleterious Effects on Exposed Surfaces

- Metal Flashing, Galvanized, Copper, others



TEST AND EVALUATION FINAL REPORT

Prototype Shingle Rejuvenators

July 7, 2021

Report For: Soylei Innovations, LLC
812 Ashworth Road
West Des Moines, IA 50265

Attn: Michael J. Forrester

Email: Michael.j.forrester91@gmail.com

Sample Data / Information:

SAMPLE ID	MATERIAL TYPE	SOURCE	RECEIVED DATE	PRI PROJECT #
Aged Shingles	Architectural	Clearwater, Home	12/2020	SOYL 01-02-01/03
SoyLei A	Prototype Rejuvenator	SoyLei Innovations	04/26/21	
SoyLei B				
SoyLei C				

OBJECTIVE: Evaluate prototype shingle rejuvenators as received at an application rate of 200 ft² per gallon for selected properties

CONCLUSIONS:

The Table below provides a comparative ranking of the treated shingles for the selected properties, with 1 = Best and 3 = Worst with respect to change when compared to the Control without rejuvenator treatment

Table 1: Comparative Rankings by Selected Property – Revised

PROPERTIES			TEST METHODS	RESULTS, SAMPLE ID, Prototype Treatment			
				SoyLei A	SoyLei B	SoyLei C	
Treated Shingles							
Color		Initial	23±2°C	Gray Scale, L	1	2	3
		1 Week			1	2	3
		Δ			1	2	3
Granule Adhesion, g loss		Dry	23±2°C	D4977	2	1	3
		Wet			3	1	2
Tear Strength, g		MD	23±2°C	D228-13 / D1922	2	1	3
		CD			2	1	3
Pliability, # Pass	Weather Side Up	MD	23±2°C	D3462	2	2	1
		CD					
	Weatherside Down	MD					
		CD					
Fire Resistance; maximum spread, inches			UL 790	1	2	3	
Hail Impact Resistance, shingle location of drop		Joint	FM 4473M / UL 2218	1	2	3	
		Single Layer					
		Single Lap					
		Single Edge					
		Double Layer					
		Double Lab					
		Double Edge					
Comparative Ranking, Totals (lower # is Best)				16	16	26	



CONCLUSION: continued - Notes:

Color: we assumed the greater the change in color the less desirable. However, this may not be the case for every home owner. Since the aged shingles were a shade of gray, we presented the data of the L component of the CIELAB color scale. See below

Hunter L, a, b and CIE 1976 L*a*b* (CIELAB) are both **color scales** based on the Opponent-Color Theory. This theory assumes that the receptors in the human eye perceive **color** as the following pairs of opposites. **L scale:** Light vs. dark where a low number (0-50) indicates dark and a high number (51-100) indicates light. A and b are the four unique colors of red, green, blue and yellow.

Granule Loss: Granules provide protection of the asphalt coating from the Sun and Rain and are key to fire and hail impact resistances. An improvement (less loss) of granules is deemed a significant benefit.

Tear Strength: is an ASTM D3462 requirement and is primarily controlled by the fiberglass mat's weight and overall strength. Overall tear strengths were decreased by the treatments for all three prototypes. The hypothesis was based on a rejuvenator that achieved penetration to the FG mat and improved the asphalt coating adhesion to the FG fibers.

Pliability: in the first pliability test all values meet the ASTM criteria, all Passed. In a second evaluation we used the standard mandrel bend test. The methodology used lowered the temperature of the specimen and mandrel 5°F in increments until cracking was observed in 3 or more replicates run at each temperature. The results were very significant and a distinguishable benefit

Fire Resistance: there was a significant benefit of the treatments in the Spread of Flame Test. However, all four (4) cases met the UL Requirement of Class A. See Appendix for summary photos

Hail Impact Resistance: the treatments had minimal if any positive benefit. See Appendix for photos

DATA / RESULTS:

Table 2: Select Properties of Liquid Rejuvenators As Received

PROPERTIES		TEST METHODS	RESULTS, SAMPLE ID		
			SoyLei A	SoyLei B	SoyLei C
SAMPLE AS-RECEIVED					
Specific Gravity	15.6°C	D6937	0.9948	0.9865	0.9983
Total Acid Number (TAN)		D664	0.85	1.42	0.75
Solubility in H2O, %		D2042	99.35	98.90	93.20
Rotational Viscosity, cPs	30°C	D4402	35.2	36.0	204.8
	40°C		32.8	32.8	181.6
	50°C		30.4	30.4	166.6



Table 3: Selected Properties of Treated Shingles – Pliability Revised

PROPERTIES			TEST METHODS	RESULTS, SAMPLE ID			
				Control	SoyLei A	SoyLei B	SoyLei C
Treated Shingles							
Color	Initial	23±2°C	Gray Scale, L	33.4	33.6	34.9	47.6
	1 Week			32.2	32.4	28.8	40.3
	Δ			-1.2	-1.2	-6.1	-7.3
Granule Adhesion, g loss	Dry	23±2°C	D4977	3.433	3.167	1.528	4.068
	Wet			3.343	3.309	3.004	3.159
Tear Strength, g	MD	23±2°C	D228-13 / D1922	659	554	640	461
	CD			586	461	566	438
Pliability, temperature at failure 1" diameter mandrel, 180° in 2 sec		75°F	D5147	Pass	Pass	Pass	Pass
		65°F		Pass	Pass	Pass	Pass
		55°F		Fail	Pass	Pass	Pass
		45°F		-	Fail	Fail	Pass
		35°F		-	-	-	Fail
Fire Resistance; maximum spread, inches			UL 790	44	14	28	29
Hail Impact Resistance, shingle location of drop	Joint		FM 4473M / UL 2218	Fail	Fail	Fail	Fail
	Single Layer						
	Single Lap						
	Single Edge						
	Double Layer						
	Double Lap						
	Double Edge						

DISCUSSIONS: Data highlighted indicates the 'Best' when compared to all 3 samples + the Control

Color: Sample A's color remained about the same. It is assumed the bigger the change in color the more the homeowners may object?

Granules Adhesion: Sample B exhibited the least granule loss in both the Dry and Wet conditions. Improved adhesion provides multiple benefits: improved fire and hail impact damage and reduced aging due to reduced asphalt exposure to UV and rain

Tear Strength: Tear strength is correlated to coating embrittlement, the rejuvenators softened the coating. The softening resulted in reduce Tear Strengths

Pliability: Sample C exhibited excellent results. A 5-10°F reduction in temperature is considered excellent Sample C's cold temperature is better than most shingles when produced. A temperature of 50 – 55 °F is considered excellent.

Fire Resistance: this is a significant and important finding. If the FR lasts, it has potential impact / benefit outside of rejuvenation

Hail Impact: results were mixed.



RECOMMENDATIONS:

1. determine the life-cycle of the treatment
2. Determine optimum application rates
3. Validate the Pliability findings to determine if this has efficacy with newly produced shingles
4. Determine if a blend of A & C provides FR & Pliability benefits with a minimum color change
5. Determine if Hail Impact Resistance in part is a function age after treatment.

NEXT STEP: review by SoyLei

APPENDIX:

Tested by:


Greg Lavin, Laboratory Supervisor

Date: July 7, 2021

Reviewed by:

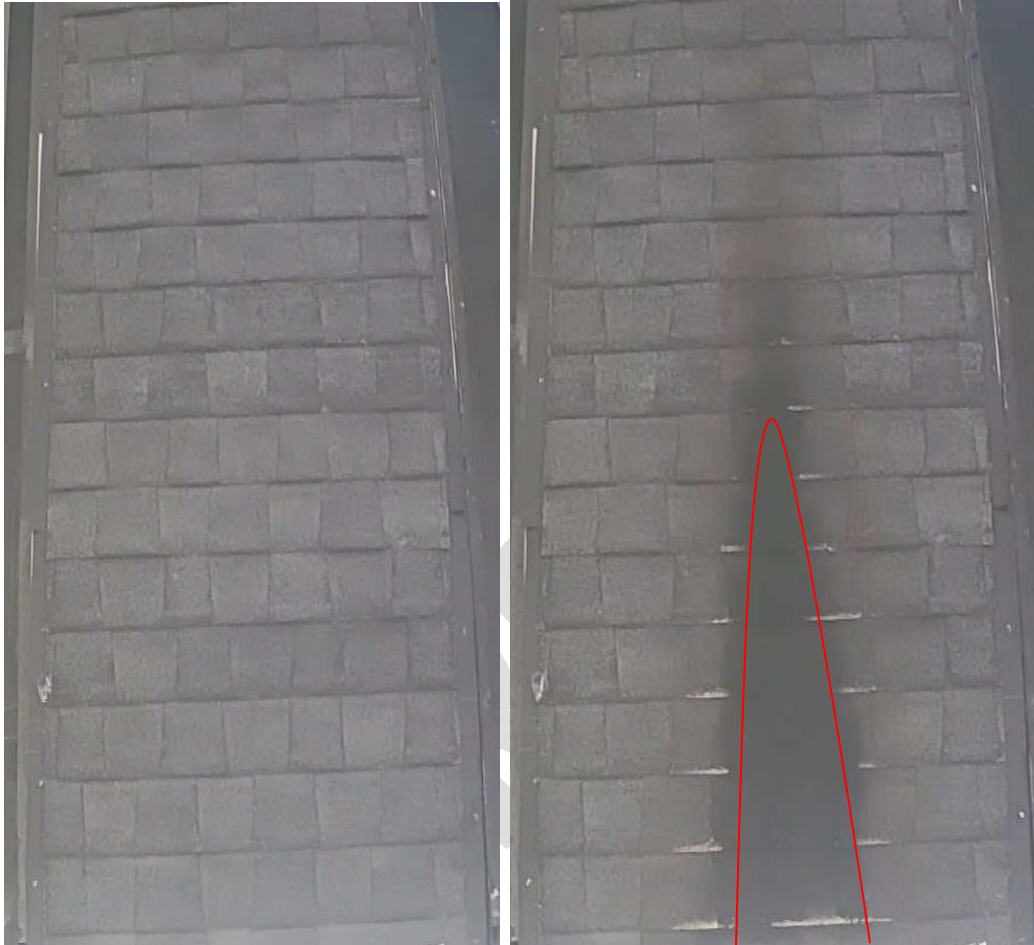

Ken Grzybowski, President

Date: July 7, 2021



APPENDIX

Control Deck – Spread of Flame Test



Before

After

Class A Testing:

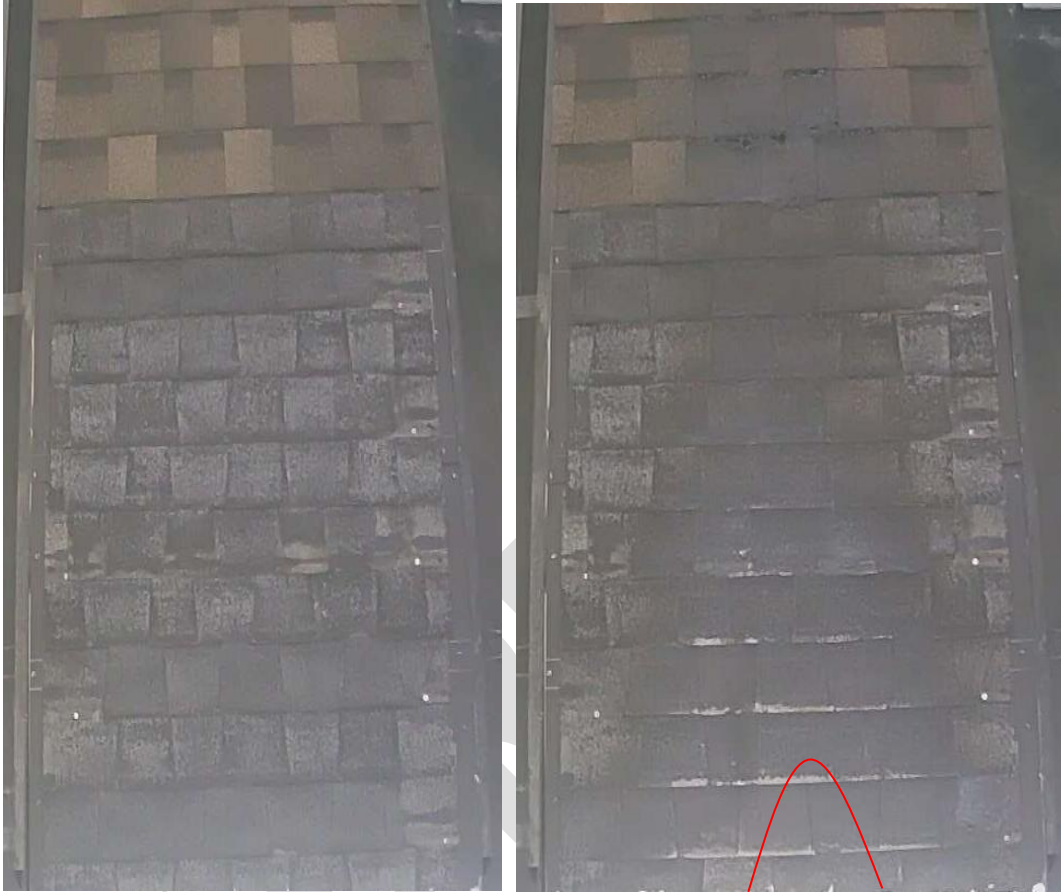
1,400°F
Flame on for 10 minutes
Burn length 72 inches or less
Deck 8' x 3.3' / Pitch 5:12
As per ASTM E108

Control Deck burned 44" passes a Class A



APPENDIX

Deck A – Spread of Flame Test



Before

After

Spread of Flame Test – Deck A, applied at 200 ft²/gal (13.5 ft², 8.64 oz. applied)

Class A Testing:

1,400°F

Flame on for 10 minutes

Burn length 72 inches or less

Deck 8' x 3.3' / Pitch 5:12

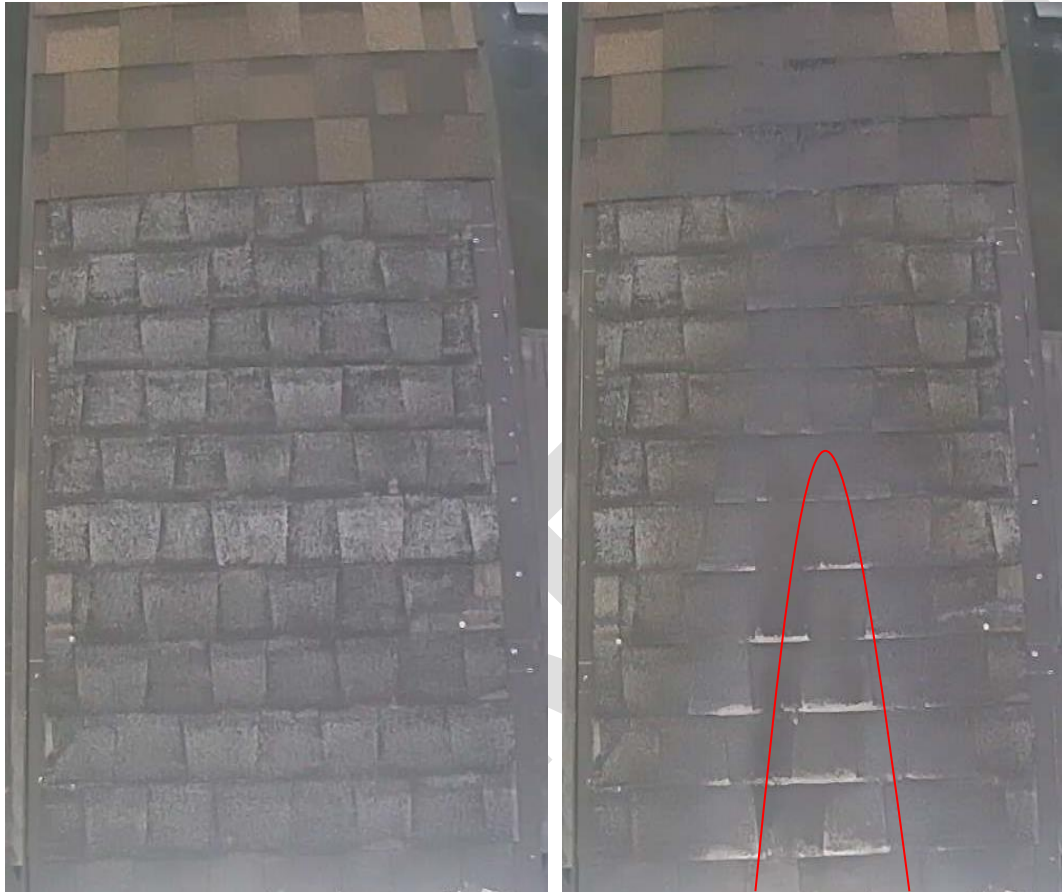
As per ASTM E108

Deck A burned 14" passes a Class A, burned 30" less than control



APPENDIX

Deck B – Spread of Flame Test



Before

After

Spread of Flame Test – Deck B, applied at 200 ft²/gal (13.5 ft², 8.64 oz. applied)

Class A Testing:

1,400°F

Flame on for 10 minutes

Burn length 72 inches or less

Deck 8' x 3.3' / Pitch 5:12

As per ASTM E108

Deck B burned 28" passes a Class A, burned 16" less than control

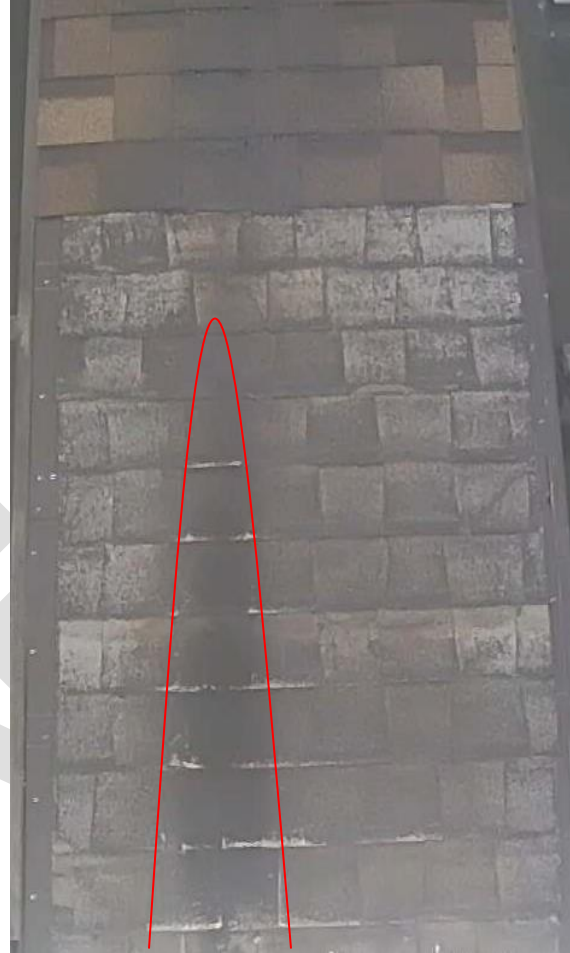


APPENDIX

Deck C – Spread of Flame Test



Before



After

Spread of Flame Test – Deck C applied at 200 ft²/gal (13.5 ft², 8.64 oz. applied)

Class A Testing:

1,400°F

Flame on for 10 minutes

Burn length 72 inches or less

Deck 8' x 3.3' / Pitch 5:12

As per ASTM E108

Deck C burned 29" passes a Class A, burned 15" less than control



APPENDIX

Hail Impact Resistance



Control Deck Mark Up



Deck A Mark Up



Deck B Mark Up



Deck C Mark Up



APPENDIX

Hail Impact Resistance

Control Deck



Fail -
Cracking

Control Deck Hail Test Double Edge



Fail -
Cracking

Control Deck Hail Test Single Lap



Pass – No
Cracking

Control Deck Hail Test Double Field



APPENDIX

Hail Impact Resistance



Fail -
Cracking

Deck A Hail Test Double Edge



Fail -
Cracking

Deck A Hail Test Single Lap



Pass – No
Cracking

Deck A Hail Test Double Field



APPENDIX

Hail Impact Resistance



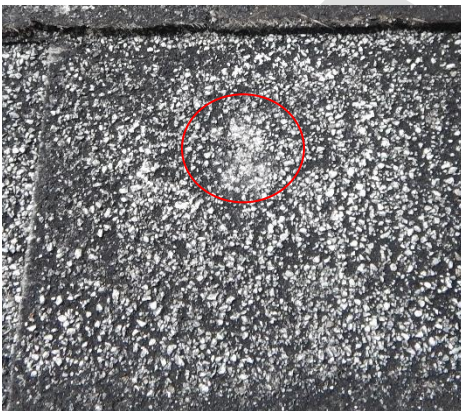
Fail -
Cracking

Deck B Hail Test Double Edge



Fail -
Cracking

Deck B Hail Test Single Field



Fail -
Cracking

Deck B Hail Test Double Field



APPENDIX

Hail Impact Resistance



Fail -
Broke

Deck C Hail Test Double Edge



Fail -
Cracking

Deck C Hail Test Single Lap



Fail -
Cracking

Deck C Hail Test Double Field

CONSULTING SERVICES**APPENDIX**

TEST & EVALUATION REPORT
(Testing & Evaluations Performed by PRI Asphalt Technologies)
Shingle Rejuvenator Study

November 10, 2024

Report For: SoyLei Innovative Products

Attn: Austin D. Hoffman, Ph. D.

Email: austin.hohmann@soylei.com

Data / Information:

SAMPLE ID	GRADE/TYPE	DATE SAMPLED	DATE RECEIVED	SOURCE
Aged Asphalt Shingles *	Architectural – Post Consumer	*Note 1	9/15/22	Roofing Contractor
Shingle Rejuvenator	"A"	---	8/21/24	SoyLei Innovations, LLC
Shingle Rejuvenator	"B"			
Shingle Rejuvenator	"C"			

**Shingles removed from a home in Clearwater, FL approximately 13 years after installation*

OBJECTIVES:

Conduct a study of selected prototype asphalt shingle rejuvenators, utilizing aged asphalt shingles that were removed from a home after approximately 13 years of exposure in Clearwater, Florida.

Determine and quantify the selected performance benefits of the aged shingles made by the rejuvenator application. Compare and quantify the differences to Control shingles, untreated set of shingles.

The study used miniature steep sloped roofs that simulated actual application parameters of standard roofs.

Construction was at PRI made with commonly used stock material (2X4's, plywood, peel-n-stick underlayment, and stainless-steel roofing nails).

All testing decks were roofed with the aged shingles, and then treated with rejuvenator according to the manufacturer's recommendations (2:1 Dilution rate applied at 0.005 gallons per square foot).

The selected performance properties were based on key physical properties that improved the shingles overall durability / life cycle while maintaining environmental compliances', color / appearance, granule adhesion, tensile strength, flexibility / pliability, hail impact resistance and resistance to spread of flame and burning brand

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Shingle Rejuvenator Study
November 10, 2024 Page 2 of 19

CONCLUSIONS:

- The rejuvenators gave no indication of detrimental environmental effects (EPA 24, Volatiles, Chlorides, Sulfates, PAH's, PCB's, Metals and pH). Testing was performed on the leachate via simulated rain using deionized water
 - Only sample B was tested.
 - Additionally, no substances of environmental concern were found when testing the rejuvenator directly.
- The application of rejuvenator causes slight, but notable changes to the shingle color. This may be an aesthetic plus
- Both Pliability / Flexibility and granule adhesion was improved

Note: these benefits indicated the rejuvenator application is improving the shingles durability / life cycle. As shingle asphalt oxidatively ages it becomes more brittle and loses granule adhesion. This typically results in reduced wind, hail and fire resistance.

- The addition of rejuvenators had no significant impact on fire, and hail resistance after application.

RECOMMENDATIONS:

Determine the benefits / life cycle of the rejuvenators at selected application rates – determine time for re-application

- Confirm environmental properties for Rejuvenators A & C.
- Determine life cycle of rejuvenator after aging, including:
 - Granule loss during exposure
 - Color differences compared to un-treated shingles during and after exposure.
 - Pliability and Flexibility after Accelerated Weathering.
 - Impact Resistance after Accelerated Weathering.
 - Environmental Assessment of 'Rain' Runoff after Application and accelerated weathering

NEXT STEPS:

- Review by Client

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DATA / RESULTS (Environmental Properties):

PROPERTIES	TEST METHODS	RESULTS, REJUVENATOR ID		
		A	B	C
Leachates As-Collected				
Select Environmental Properties				
Leachate collection of runoff water	PRI	Not Tested	√	Not Tested
EPA 24 Volatiles, %	EPA 24		0.02	
EPA 24 Volatiles, g/L			0.20	
Chlorides	EPA 365-3		BDL	
Sulfates	EPA 375-4		BDL	
Total PAHs, ppm	EPA 8270		BDL	
Total PCBs, ppm	EPA 8082		BDL	
Metals Content, ppm	EPA 6010		57.5	
pH	EPA 9040		6.6	

BDL – Below Detectable Limit

Note – Full Breakdown of Environmental Data 7 Individual Compounds in Appendix A-1 through A-4

DISCUSSION: Lab Roof Deck Preparation Protocol

A small (2'x2') roof deck was made for each rejuvenator (only sample “B” tested initially per client request).

The respective rejuvenators were applied to the roof decks at a 2:1 dilution ratio, at 0.005 gal/sq. ft.

The samples were then allowed to cure outdoor in dry conditions for 72 hours before testing.

The samples were then placed into PRI's APWS, Asphalt Pavement and/or Roofing Weathering System for 100 hours,

The water draining from the sample area was collected over the course of the first 100 hours for the testing of environmental impact properties.

The tested sample did not exhibit problematic amounts of any of the EPA targeted compounds (Note: Control sample was not tested).

See table above and appendix for results.

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DATA / RESULTS (Physical Properties):

PROPERTIES		TEST METHODS	RESULTS, REJUVENATOR ID			
			Control	A	B	C
Shingle Samples As-Treated						
Select Environmental Properties						
Color; CEILAB color scale	L	E1347	36.13	26.90	30.11	32.98
	a		0.03	0.46	-0.11	0.19
	b		3.84	1.30	1.84	4.10
	ΔE		N/A	9.59	6.35	3.17
	Visual	PRI				
Granular Adhesion, % Loss		D4977	13.2	8.0	7.8	7.1
Tear Strength (Machine Direction), N		D288	4.3	5.0	4.9	5.1
Tear Strength (Cross Direction), N			3.9	4.5	4.5	4.5
Pliability, °C		D3462	-3	-6	-6	-6

DISCUSSION:

A small (2'x3') roof deck was made for each rejuvenator. The respective rejuvenators were applied to the roof decks at a 2:1 dilution ratio, at 0.005 gal/sq. ft. The samples were then allowed to cure outdoor in dry conditions for 72 hours before testing.

See table above for results

L = perceptual lightness, 0 is pure black, 100 is pure white

a & b = four unique colors of human vision: red, green, blue and yellow

The rejuvenator application tended to make the shingles whiter, restoring to a degree the original color of the shingles

The Control shingles exhibited a granule loss of $\approx 13.2\%$. the rejuvenator treated shingles exhibited a significant reduction in granule loss. Granule retention significantly impacts a hingles durability and resistances to Wind, Fire, and Hail.

Tear and Tensile Strengths are used to define a shingle's overall strength and durability. The rejuvenator treated shingles all exhibited increases in granule adhesion, Tear and Tensile strengths. The rejuvenator treatment is to an extent restoring the shingles overall strengths.

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DATA / RESULTS (Spread of Flame):

PROPERTY		TEST METHODS	RESULTS, SAMPLE ID			
			Control	A	B	C
Select Properties						
Resistance to Flame – Spread of Flame	Max Allowed Spread, in	E108	72	72	72	72
	Observed Spread, in		53	38	44	34
	Time to ignition, mins : secs		2:05	1:21	1:13	4:52
	Pass/Fail		Pass	Pass	Pass	Pass

DISCUSSION:

At no time during or after testing were any of the following observed: roof cover or roof deck material blowing/falling off of the test deck in the form of flaming or glowing brands or particles that continue to glow after reaching the floor, the combustible roof deck exposed, lateral spread of flame, or spread of flame beyond 6 ft. (Class A), 8 ft. (Class B), or 13 ft. (Class C).

The addition of the rejuvenators to the roof decks slightly reduces the spread of flame. All but the sample treated with Rejuvenator C reduce the time to ignition, but not enough to cause the shingles to fail to meet the Class A requirements.

DATA / RESULTS (Burning Brand):

PROPERTY		TEST METHODS	RESULTS, SAMPLE ID			
			Control	A	B	C
Select Properties						
Resistance to Flame – Burning Brand	Flames 2ft. above brand, mins:secs	E108	0:41	0:39	1:01	0:43
	Smoke on Underside, mins:secs		4:38	4:02	3:16	4:37
	Discoloration on Underside, mins:secs		6:25	4:57	6:22	5:28
	Flames on Underside, mins:secs		33:30	31:54	43:48	22:43
	Pass/Fail		Fail	Fail	Fail	Fail

DISCUSSION:

All four of the tested decks failed the Burning Brand test. With all four decks sustained flames were observed on the underside of the roof deck during testing.

The addition of the rejuvenators to the roof decks appears to have a negligible effect on the results of the burning brand test.

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DATA / RESULTS (Hail Impact):

PROPERTY		TEST METHODS	RESULTS, SAMPLE ID			
			Control	A	B	C
Select Properties						
Hail Impact Resistance	Average Ice Ball Mass, g	FM4473	42.9	43.1	43.3	43.5
	Average Velocity, mph		67.7	67.9	67.8	67.8
	Average Kinetic Energy, ft-lb.		14.5	14.7	14.7	14.7
	Pass/Fail		Fail	Fail	Fail	Fail

DISCUSSION:

At the treatment level selected all test specimens exhibited cracking, breakage, and/or dis-bonding when subjected to hail impact testing. No discernable difference was observed with the application of the rejuvenators.

A number of options exist to improve the benefits of SoyLei's rejuvenator;

1. Treat the shingles earlier in the life-cycle
2. Increase the concentration of active ingredients

Tested by:


Greg Lavin, Quality Assurance Manager

Date: November 10, 2024

Reported by:


Steven Loeffler, Client Services Manager

Date: November 10, 2024

Reviewed by:

Ken Grzybowski, Consultant

Date: November 10, 2024

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APPENDIX A-1 (Full PCB Data):

PROPERTY		TEST METHOD	METHOD DETECTION LIMIT (ppm)	RESULTS, SAMPLE ID		
				A	B	C
Leachates As-Collected						
Select Requirements						
PCB, ppm	PCB-1016 (Aroclor 1016)	EPA 8082	0.5	Not Tested	BDL	Not Tested
	PCB-1221 (Aroclor 1221)		0.5		BDL	
	PCB-1232 (Aroclor 1232)		0.5		BDL	
	PCB-1242 (Aroclor 1242)		0.5		BDL	
	PCB-1248 (Aroclor 1248)		0.5		BDL	
	PCB-1254 (Aroclor 1254)		0.5		BDL	
	PCB-1260 (Aroclor 1260)		0.5		BDL	

BDL: Below detectable limit

DISCUSSION: Results indicate PCB's were not detected at the limited value, 0.5 ppm

APPENDIX A-2 (Full VOC Data):

PROPERTY		TEST METHOD	RESULTS, SAMPLE ID		
			A	B	C
Leachates As-Collected					
Select Requirements					
Density, g/L	25°C	D70	Not Tested	0.9842	Not Tested
Volatile Matter, %		D2369		64.62	
Water Content, %		D95		64.60	
Exempt Solvents, %		D4457		0.00	
EPA Volatiles, %		EPA 24		0.02	
EPA Volatiles, g/L				0.20	

BDL: Below detectable limit

DISCUSSION: See the results in the above table.

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APPENDIX

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APPENDIX A-3 (Full PAH Data):

PROPERTY		TEST METHOD	DETECTION LIMIT (ppm)	RESULTS, SAMPLE ID		
				A	B	C
Leachates As-Collected						
Select Requirements						
PAH, ppm	Acenaphthene	EPA 8270C	0.05	Not Tested	BDL	Not Tested
	Acenaphthylene		0.05		BDL	
	Anthracene		0.05		BDL	
	Benzo(a)anthracene		0.05		BDL	
	Benzo(a)pyrene		0.05		BDL	
	Benzo(b)fluoranthene		0.05		BDL	
	Benzo(j)fluoranthene		0.05		BDL	
	Benzo(k)fluoranthene		0.05		BDL	
	Benzo(g,h,i)perylene		0.05		BDL	
	Chrysene		0.05		BDL	
	Dibenz(a,h)acridine		0.05		BDL	
	Dibenz(a,j)acridine		0.05		BDL	
	Dibenzo(a,h)anthracene		0.05		BDL	
	7H-Dibenzo(c,g)carbazole		0.05		BDL	
	Diabenzo(a,e)pyrene		0.05		BDL	
	Diabenzo(a,h)pyrene		0.05		BDL	
	Diabenzo(a,i)pyrene		0.05		BDL	
	Fluoranthene		0.05		BDL	
	Fluorene		0.05		BDL	
	Ideno(1,2,3-cd)pyrene		0.05		BDL	
	3-Methylcholanthrene		0.05		BDL	
	Naphthalene		0.05		BDL	
	Phenanthrene		0.05		BDL	
	Pyrene		0.05		BDL	
Total PAH Content, ppm		Calc.	---	BDL		
Total PAH Content, %		Calc.	---	BDL		

BDL: Below detectable limit

DISCUSSION: Results indicated all PAH's were below the detectable limit of 0.05 ppm

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APPENDIX A-4 (Full Metals Data):

PROPERTIES		TEST METHODS	RESULTS, SAMPLE ID		
			A	B	C
Leachates As-Collected					
Select Requirements					
Metals Content, ppm	Na	EPA 6010	Not Tested	BDL	Not Tested
	Ag			BDL	
	Al			BDL	
	B			17.0	
	Ba			BDL	
	Ca			BDL	
	Cd			BDL	
	Cr			BDL	
	Cu			BDL	
	Fe			BDL	
	K			40.5	
	Mg			BDL	
	Mn			BDL	
	Mo			BDL	
	Zn			BDL	
	V			BDL	
	Ti			BDL	
	Sn			BDL	
	Si			BDL	
	Pb			BDL	
	P			BDL	
	Ni			BDL	

BDL: Below detectable limit

DISCUSSION: All except, B and Fe were below dedicated limits.

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APPENDIX A-5 (Spread of Flame – Control)



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APPENDIX A-6 (Spread of Flame – A)



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APPENDIX A-7 (Spread of Flame – B)



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APPENDIX A-8 (Spread of Flame – C)



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APPENDIX

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APPENDIX A-9 (Burning Brand – Control)

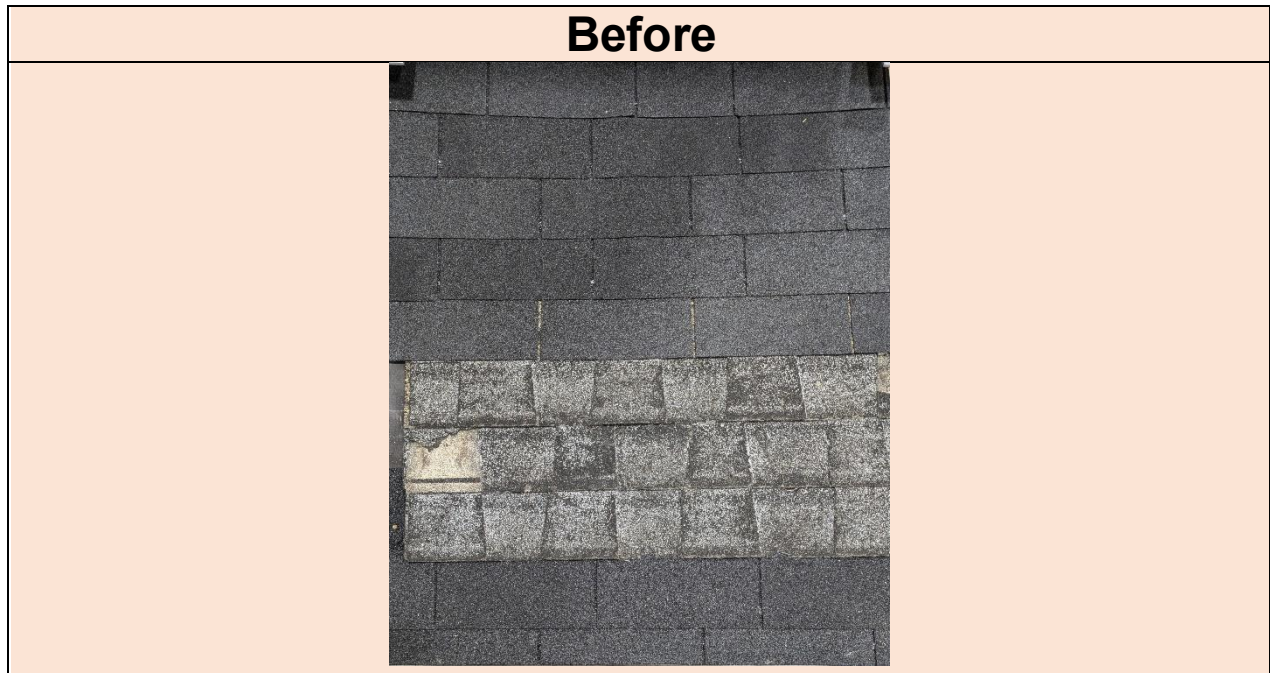


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APPENDIX

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APPENDIX A-10 (Burning Brand – A)



KFG

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APPENDIX

After



er

(Top)

After (Bottom)



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APPENDIX A-11 (Burning Brand – B)



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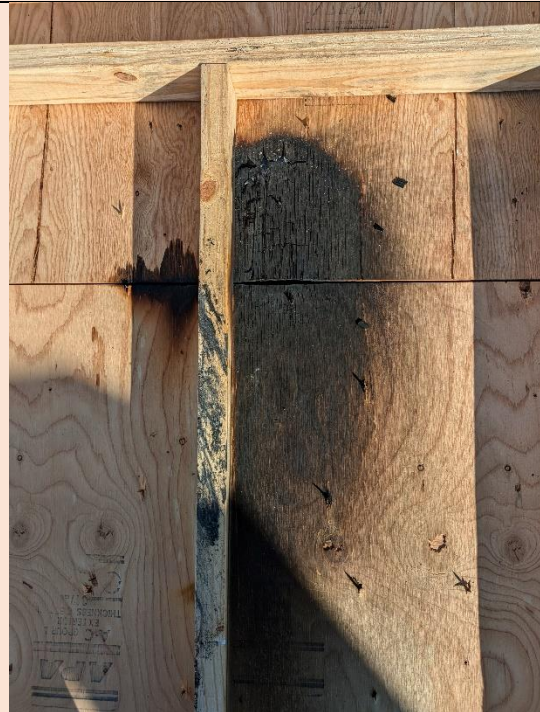
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APPENDIX A-12 (Burning Brand – C)

Before



After (Top)

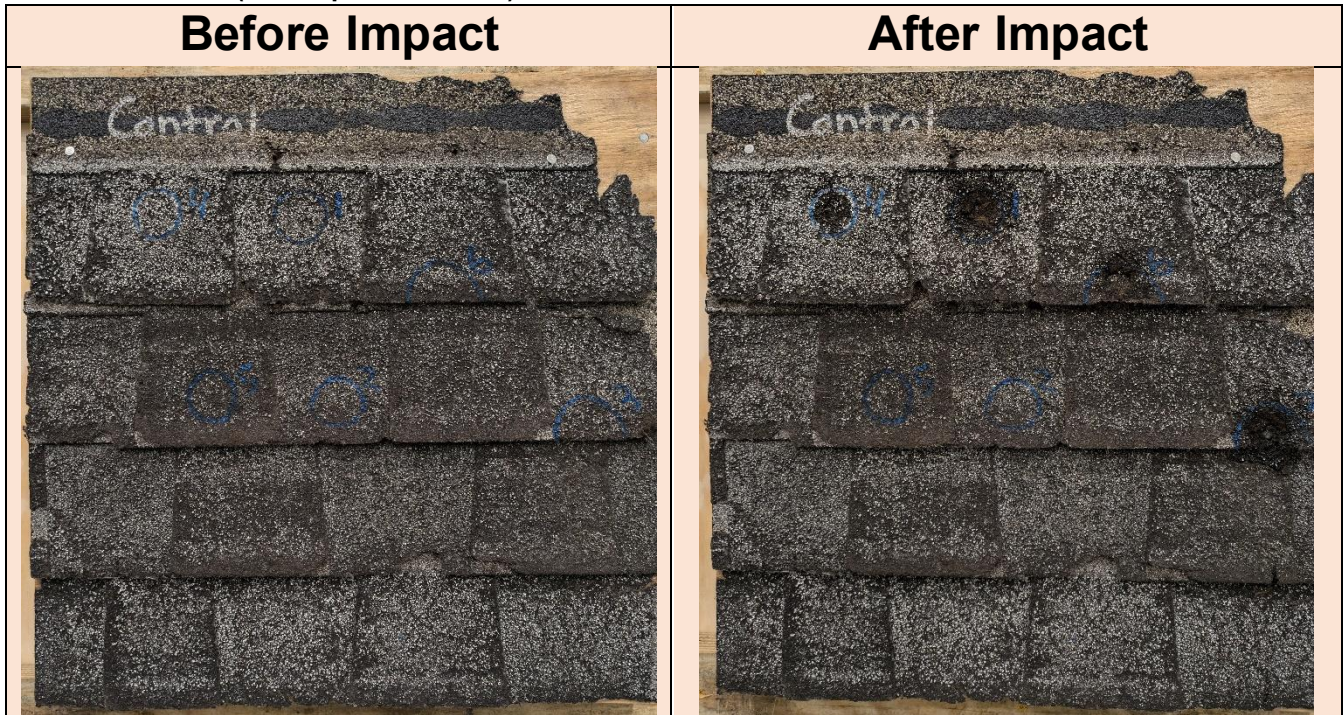


After (Bottom)

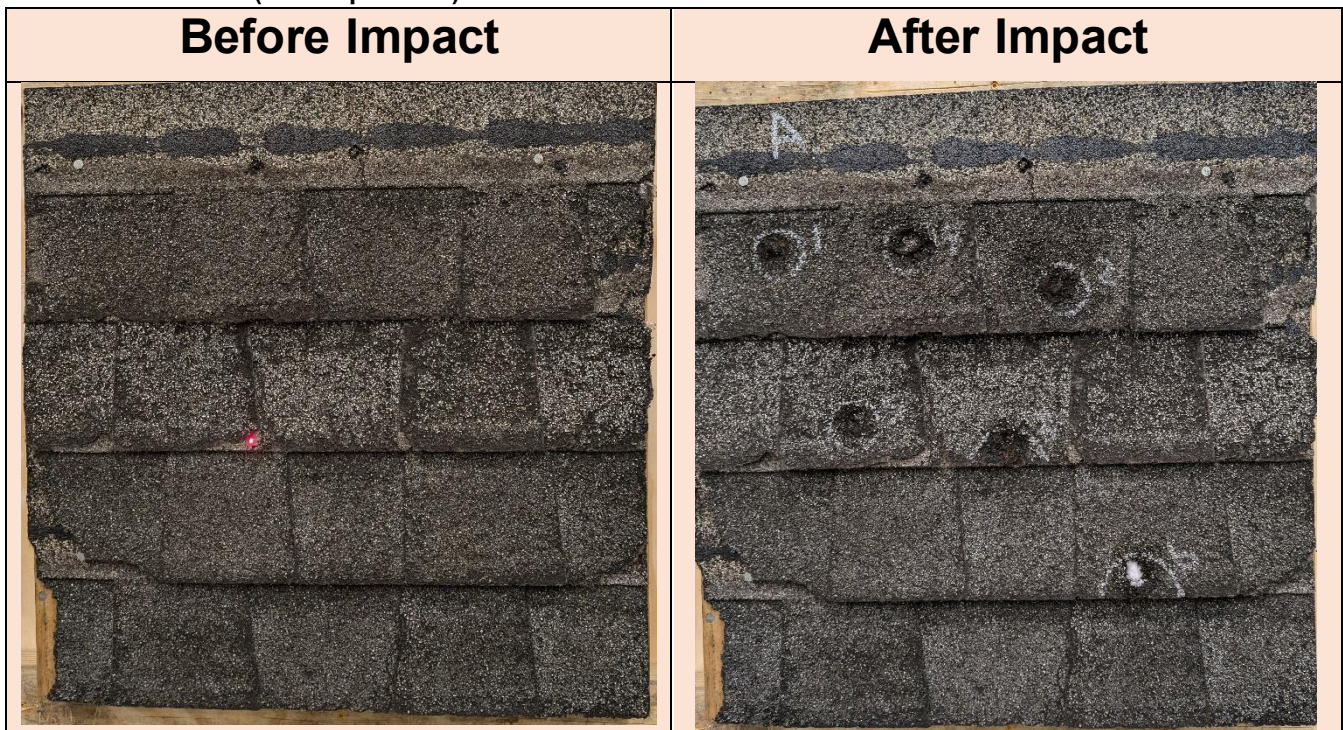
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APPENDIX

APPENDIX A-13 (Hail Impact – Control)



APPENDIX A-14 (Hail Impact – A)



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APPENDIX

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APPENDIX A-13 (Hail Impact – B)



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APPENDIX A-13 (Hail Impact – B)

APPENDIX A-14 (Hail Impact – C)

