APPLIED GRAPHENE MATERIALS

Use of Graphene Nanoplatelets to Enhance Chemical Resistance

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Enhancing Chemical Resistance

Why Graphene?

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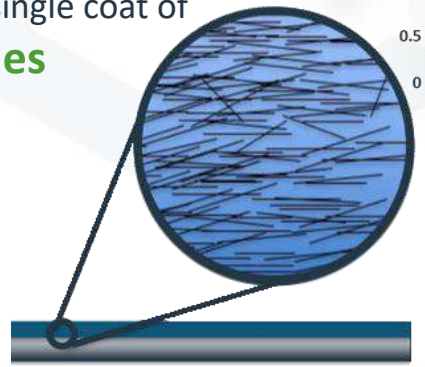
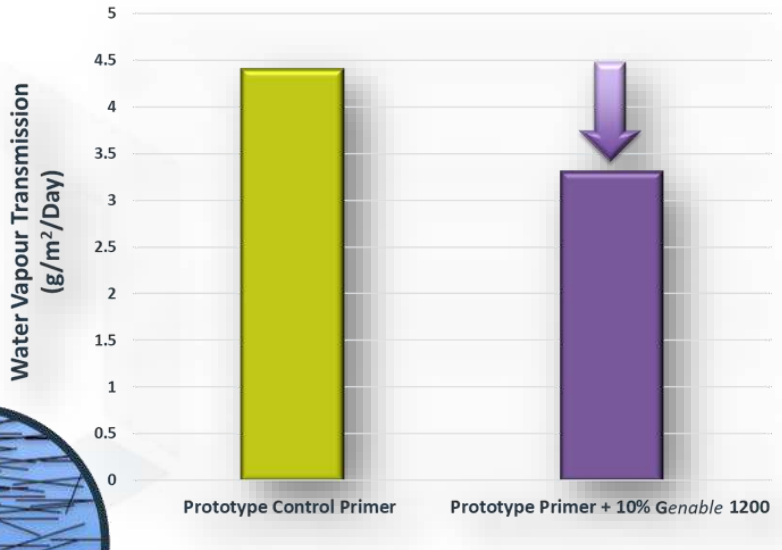
- Patent-protected technology produces few layer Graphene Nanoplatelets
- The Nanoplatelets are approximately **25,000 times** thinner than a single human hair
- The addition of just **0.1%** graphene can increase a migrating species journey through a single coat of standard industrial paint by **120 times**
- Offering outstanding barrier and anti-corrosion properties





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AGM Graphenes

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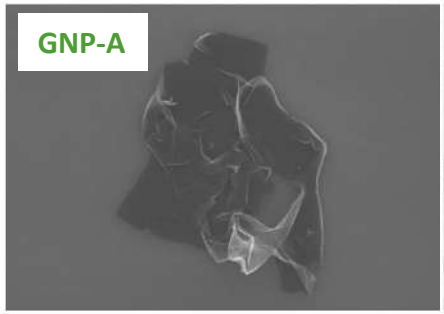

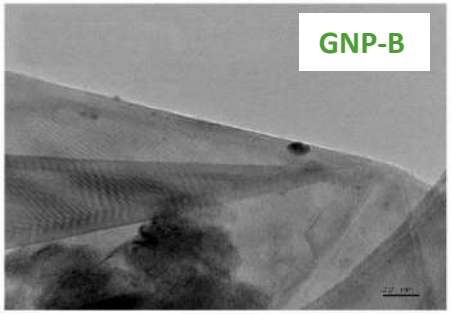

	GNP – A	GNP - B
Appearance	Black Powder	Black Powder
Particle Size (D90)(µm)	350 – 450	10 – 30
Surface Area (m2/g)	275 – 325	275 – 325
Primary Platelet Thickness	3 – 5 nm	2 – 5 nm
Tap Density (average)(g/l)	9	275 ± 70
Oxygen / Carbon Ratio	0.03 ± 0.01	0.023

- Two of AGM's graphene types were used in the tests conducted, denoted here as **GNP-A** and **GNP-B**
- The graphene types are different in morphology, synthesis route and other physical properties
- Graphene concentration loaded into dispersion:
  - **GNP-A** at **1.0%** (w%/w%)
  - **GNP-B** at **15.0%** (w%/w%)
- Graphene dispersions added to the final paint to achieve the required loading for application
- Variation in graphene loading is impacted by the high oil absorption of GNP-A and the lower density compared to GNP-B

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AGM Graphenes

- Chemical resistance is required in many applications
- Typical applications where the potential for chemical attack could arise from a range of different chemicals.
  - Flooring
  - Infrastructure, e.g. bridges
  - Secondary containment and chemical storage tanks
  - Industrial manufacturing facilities
  - Transport
  - Marine and ship
- The chemical resistance protective coatings market is set to grow by over 5% per year, reaching **$8.3 billion** by 2026

(*Analytics Market Research)*

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Experimental

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Coating Formulations

- Experiment designed to demonstrate the effect of graphene and glass flake materials on performance

Systems Tested

Glass Flake V's Graphene:

- 20.0% Glass Flake
- 0.05% GNP-A
- 0.10% GNP A
- 0.50% GNP-B
- 1.00% GNP-B

Graphene & Glass Flake Hybrids:

- 20.0% Glass Flake + 0.025% GNP-A
- 10.0% Glass Flake + 0.05% GNP-A
- 5.0% Glass Flake + 0.1% GNP-A
- 10.0% Glass Flake + 0.5% GNP-B

- Coatings produced using GNP dispersions and glass flake loadings
- Graphene dispersion in epoxy carrier resin prior to dilution into the final paint
- Cured with an Epoxy-Amine stoichiometry ratio of 85%

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Coating Formulations

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	20% Glass Flake	0.05% GNP-A	0.10% GNP-A	0.5% GNP-B	1.0% GNP-B	20% Glass Flake, 0.025% GNP-A	10% Glass Flake, 0.05% GNP-A	5% Glass Flake, 0.10% GNP-A	10% Glass Flake, 0.5% GNP-B
190 EEW Epoxy	39.83	46.04	42.28	47.42	45.03	37.95	41.06	39.79	42.43
Xylene	14.94	17.27	15.85	17.78	16.89	14.23	15.40	14.92	15.91
Butanol	4.64	5.36	4.92	5.52	5.24	4.42	4.78	4.63	4.94
Surface Wetting Agent	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
Glass Flake	20.00	-	-	-	-	20.00	10.00	5.00	10.00
GNP-A Dispersion	-	5.00	10.00	-	-	2.50	5.00	10.00	-
GNP-B Dispersion	-	-	-	3.33	6.67	-	-	-	3.33
115 AHEW Amine	20.49	26.23	26.85	25.85	26.08	20.80	25.56	20.80	23.29
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

- All figures are weight percentages

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Coating Panel Preparation

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- Duplicate panels for each coating were half-immersed in a range of standard chemical solvents
- Application by drawdown bar for a dry film thickness of 90 ± 10 microns
- All panels were cured at 25°C for 7 days
- Visual assessment after a 28-day time period for:
    - Changes in colour
    - Blistering
    - Hardness retention
    - Gloss retention
- Immersion media selected to be representative of weak and strong acids and bases as well as organic solvents

	Immersion Media
Solvent	Xylene
	Methyl Ethyl Ketone
Acids	10% Lactic Acid
	10% Sulphuric Acid
Bases	50% Sodium Hydroxide
	10% Sodium Hypochlorite

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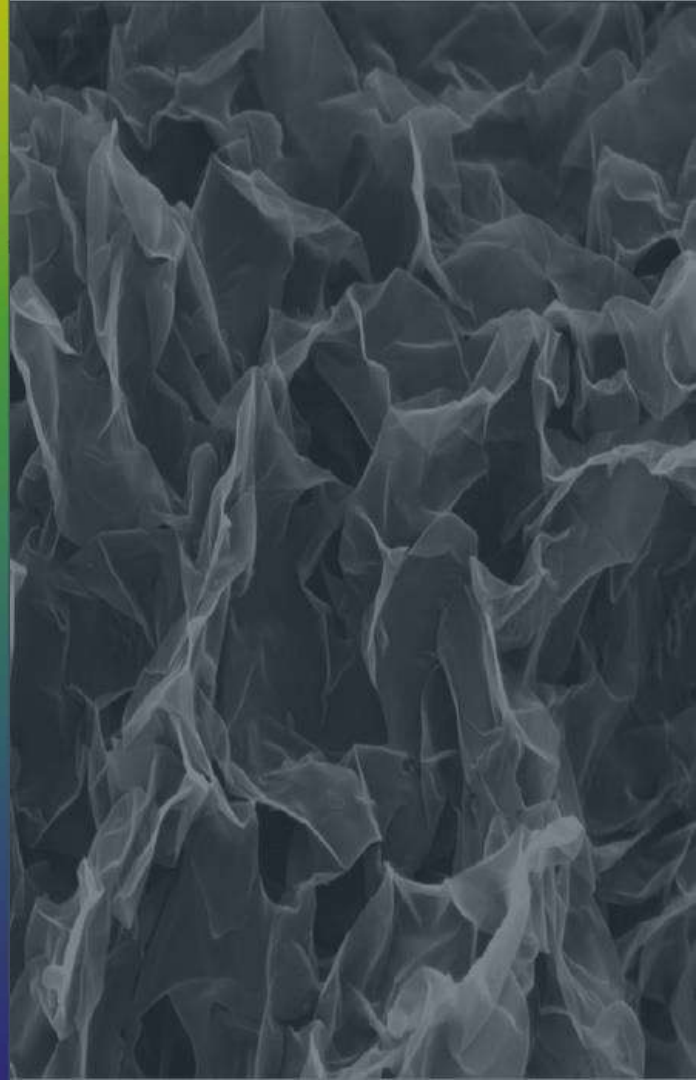
Pre-test Assessments

- Prior to testing, samples were assessed for initial gloss and hardness values.
- An unexposed sample was used to create a baseline for both gloss and hardness.
- Hardness
  - Glass Flake – 6H pencil.
  - GNP-A – H for both loadings.
  - GNP-B – F for both loadings.
  - GNP-A Hybrids – range of 4H, H and H respectively for decreasing glass flake loading.
  - GNP-B Hybrid – 3H
- Gloss @60°
  - Glass Flake – approximately 60-70 GU
  - GNP-A - approximately 95-98 GU
  - GNP-B – approximately 94-95 GU
  - GNP-A Hybrids – approximately 99, 104 and 70 GU
  - GNP-B Hybrid – approximately 100 GU



Results

Glass Flake vs. Graphene

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Xylene Resistance

- **No blistering** or surface roughening apparent on any of the test samples
- **No colour change** to the coatings at the end of the immersion period

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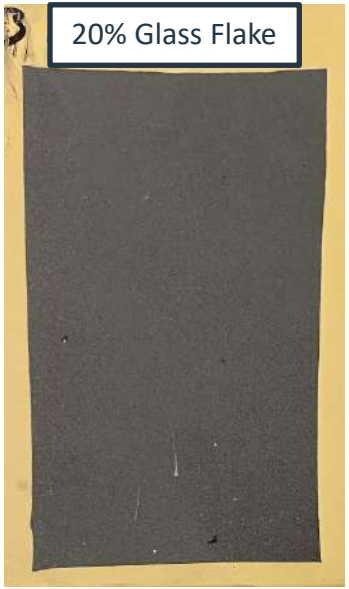

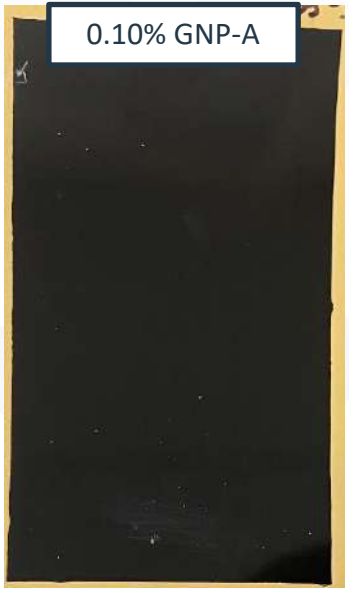

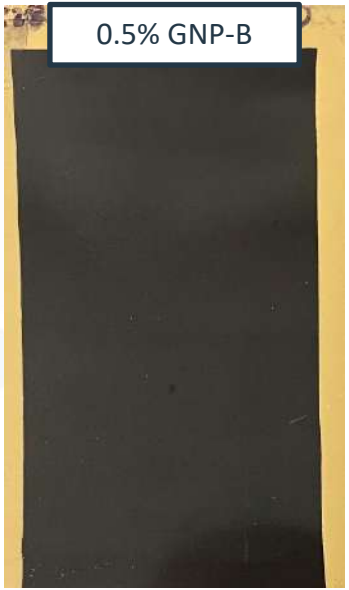

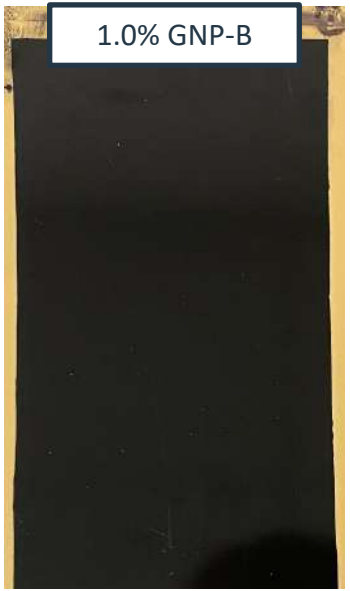

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Xylene Resistance

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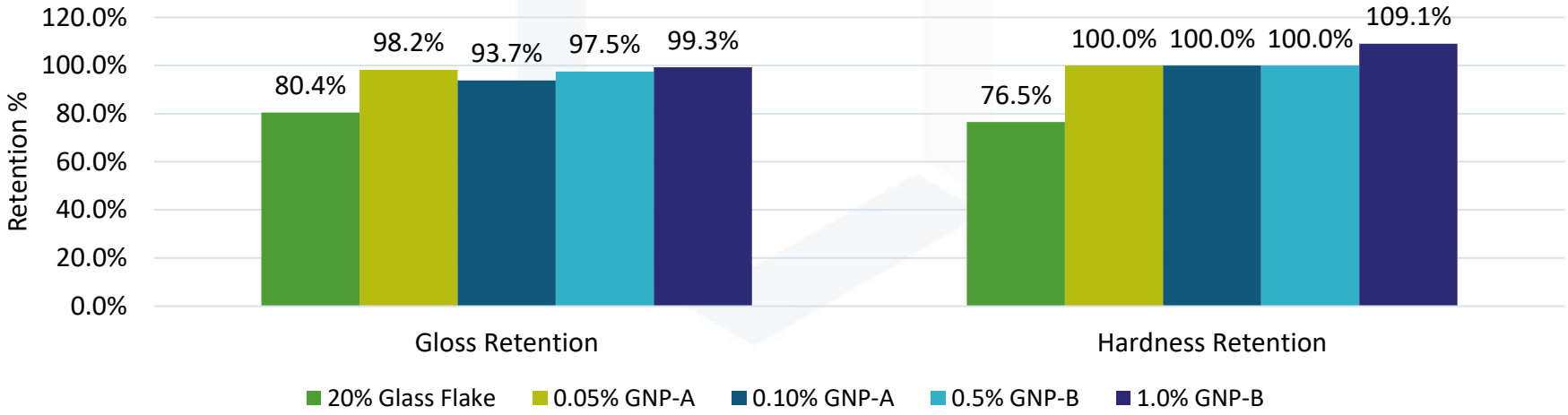
Gloss Retention:

- Retention on graphene-based coatings
  >90% for Graphene vs. 80% for glass flake coating

Hardness Retention:

- Higher levels for all graphene-containing coatings



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MEK Resistance

- **High levels of blistering** on glass flake panel - Density 4, Size 2 - Excluded from hardness and gloss testing
- **No changes** recorded on graphene-containing coatings

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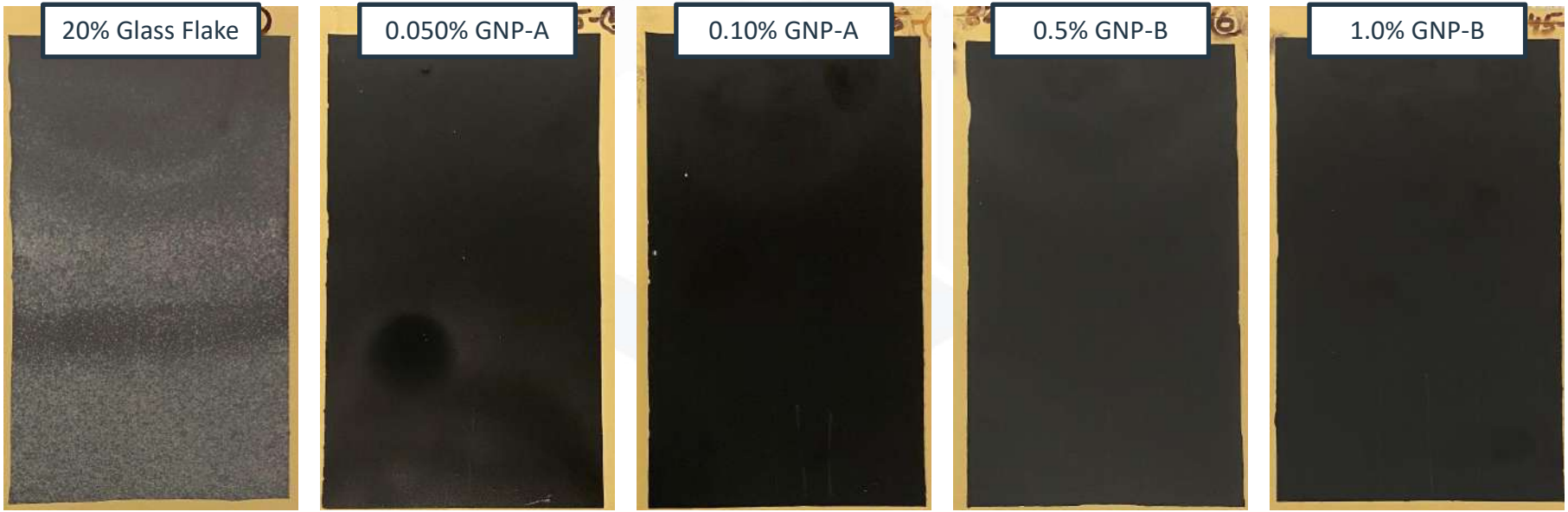


Enhancing Chemical Resistance

MEK Resistance

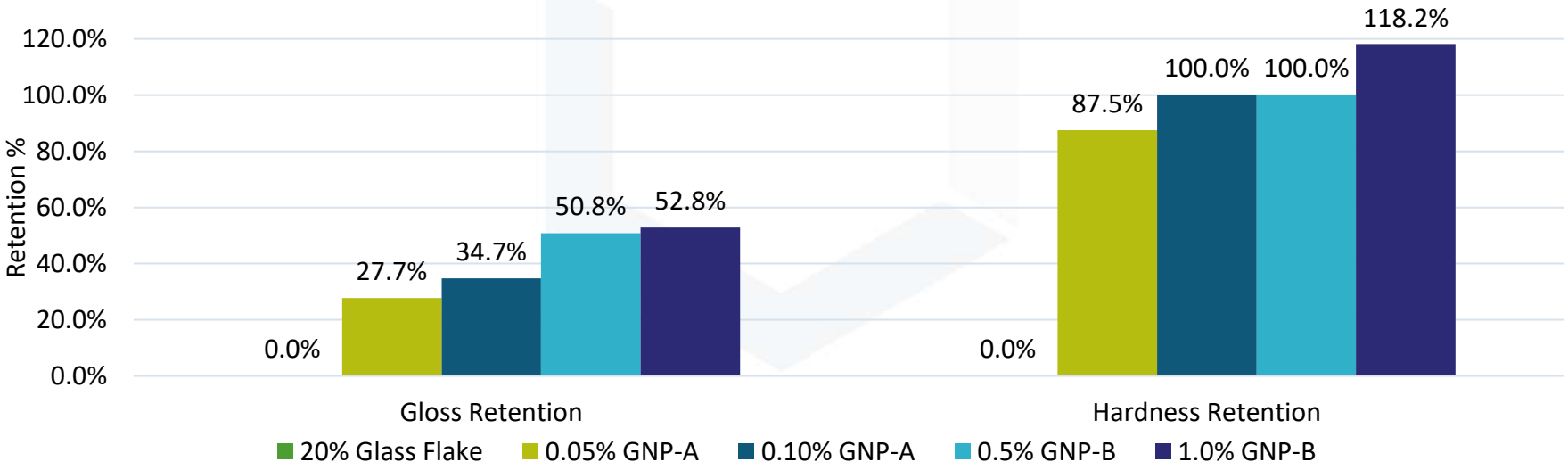
- Glass flake coating was not tested due to blistering

Gloss Retention:

- Increasing retention with loading levels on graphene-based coatings

Hardness Retention:

- Higher levels of hardness retention for all graphene-containing coatings



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Enhancing Chemical Resistance

10% Lactic Acid Resistance

- Glass flake shows significant breakdown and **failure of the coating**
- Graphene coatings show **high levels of resilience** to lactic acid exposure, **no blistering** on samples

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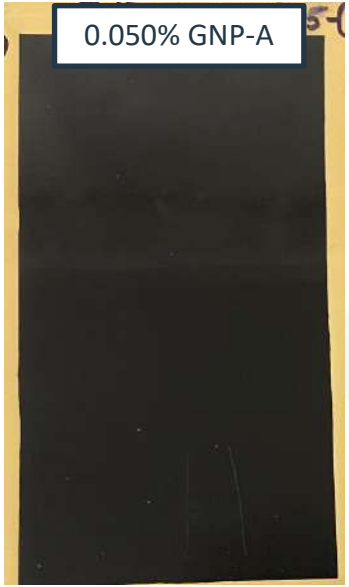


Enhancing Chemical Resistance

10% Lactic Acid Resistance

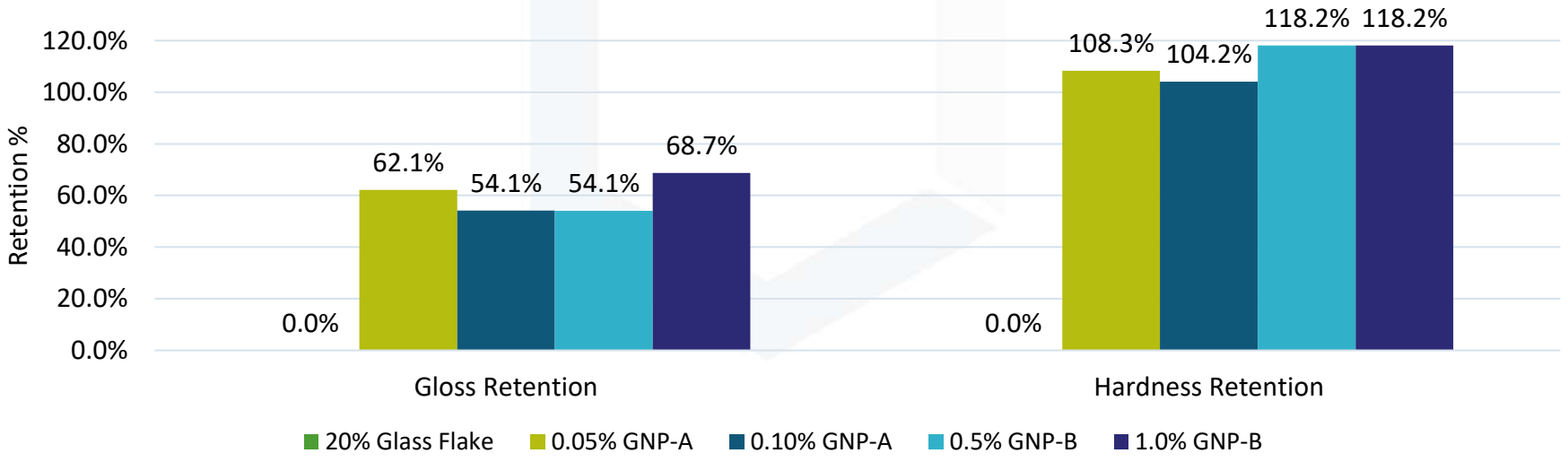
- Glass flake coating was not tested due to blistering

Gloss Retention:

- Gloss of graphene coatings above 50% of initial values

Hardness Retention:

- No loss of hardness on graphene containing coatings.



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Enhancing Chemical Resistance

10% Sulphuric Acid Resistance

- Glass flake shows significant breakdown and **failure of the coating**
- Graphene coatings show **high levels of resilience** to sulphuric acid exposure, **no blistering** on samples

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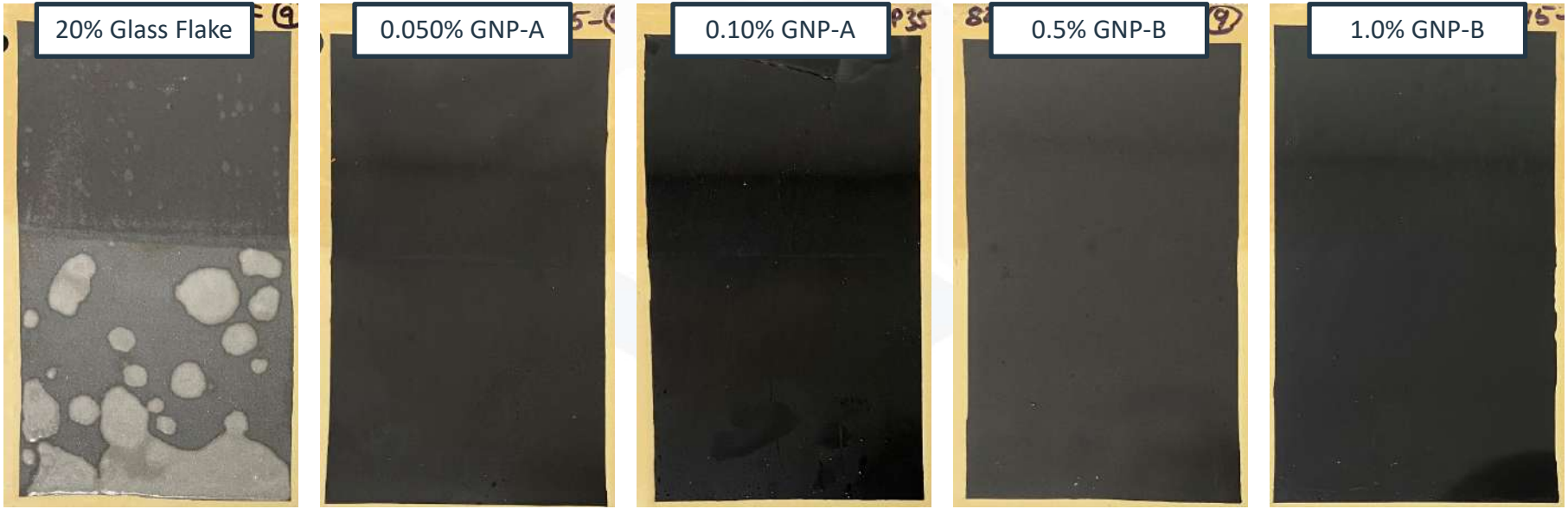


Enhancing Chemical Resistance

10% Sulphuric Acid Resistance

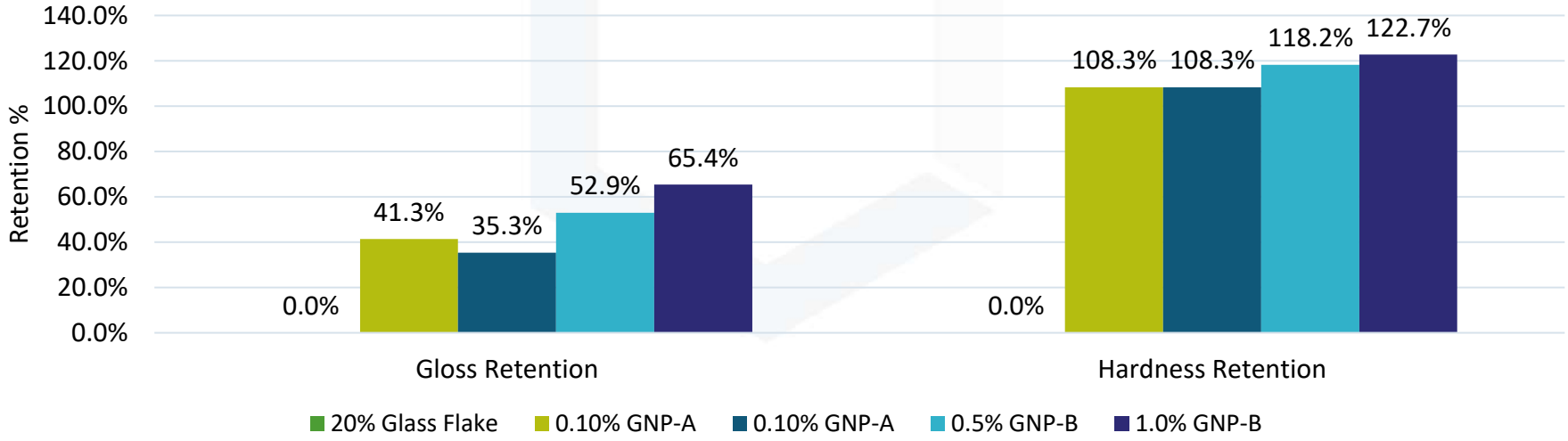
- Glass flake coating was not tested due to blistering

Gloss Retention:

- Retention varies with loading level of graphene
- GNP-B systems were the higher performing

Hardness retention:

- Graphene coatings show higher levels of retention
- Potential acid-hardening effect resulting in increases



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50% Sodium Hydroxide Resistance

- **No blistering** on any samples
- **No colour changes** were noted on any samples
- White residue is from drying, rather than effects from immersion

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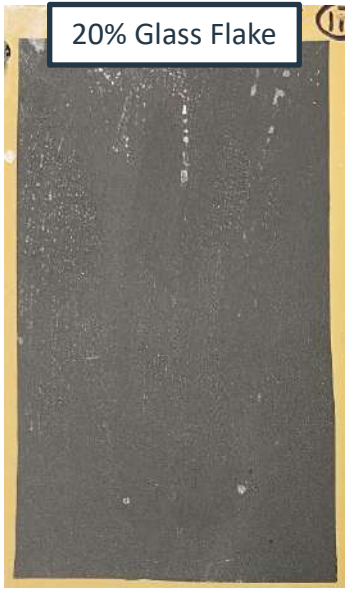

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50% Sodium Hydroxide Resistance

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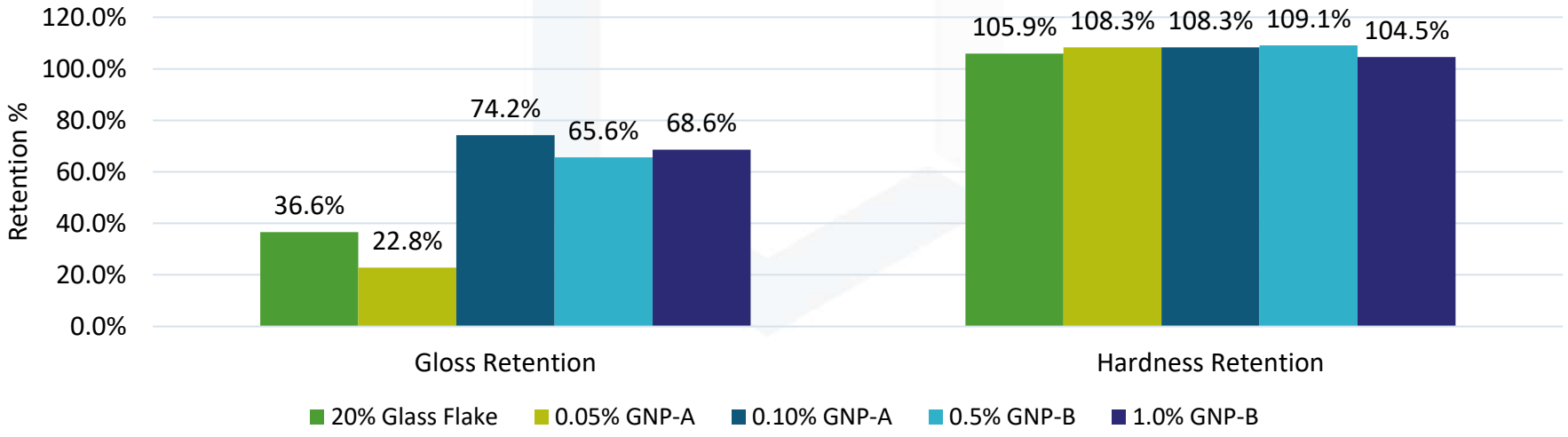
Gloss Retention:

- Low retention from glass flake and lower GNP-A
- GNP-A at a higher loading and GNP-B gave better levels of retention

Hardness retention:

- All samples showed high levels of retention in all tested systems



Enhancing Chemical Resistance

10% Sodium Hypochlorite Resistance

- **Complete failure** of the glass flake coating, rusting in immersed area
- **No visual change** for graphene coatings

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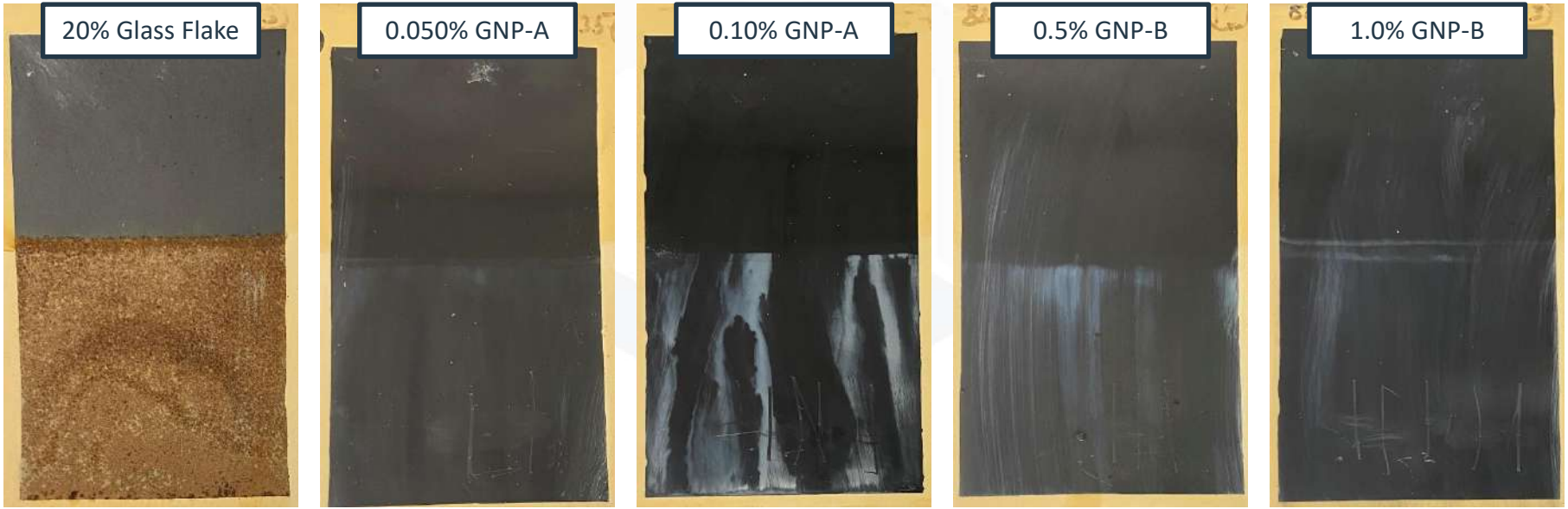


Enhancing Chemical Resistance

10% Sodium Hypochlorite Resistance

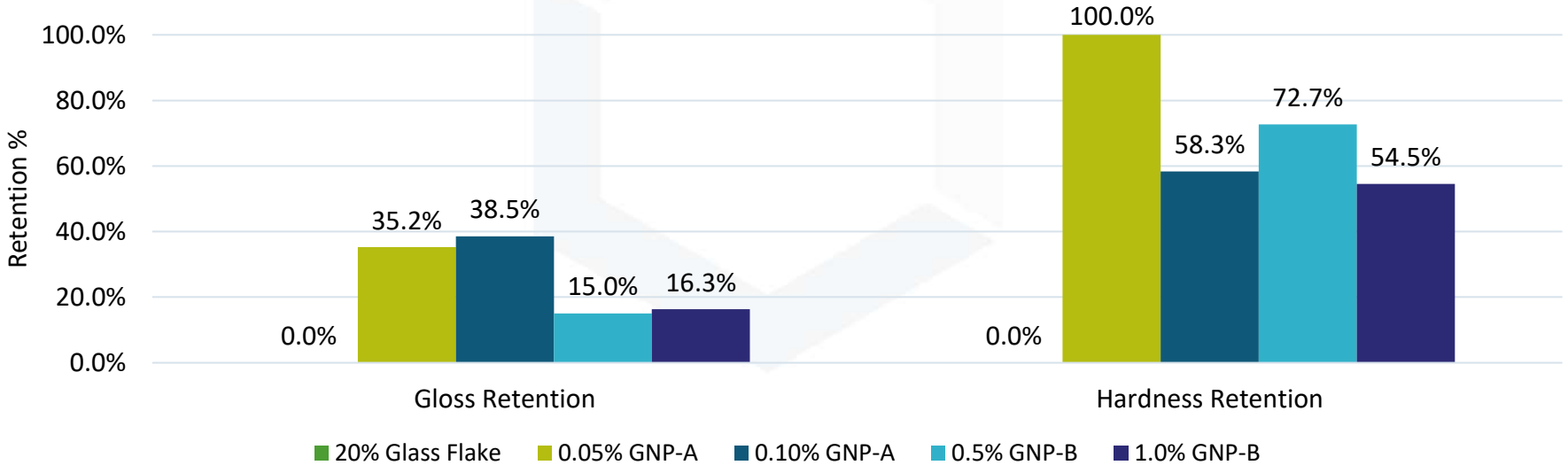
- Glass flake coating was not tested due to blistering

Gloss Retention:

- Highest retention from GNP-A
- GNP-B showed some levels of retention

Hardness retention:

- Full retention for low-loading GNP-A coating
- Other systems show some softening of coating



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Hybrid Systems

Graphene & Glass Flake Combined

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10% Lactic Acid Resistance

- **Complete coating failure** for the glass flake coating and hybrid coating with high glass flake loading
- Increasing loadings of GNP-A and decreasing loading of glass flake, **improves the coating's visual performance** and resistance to chemical ingress

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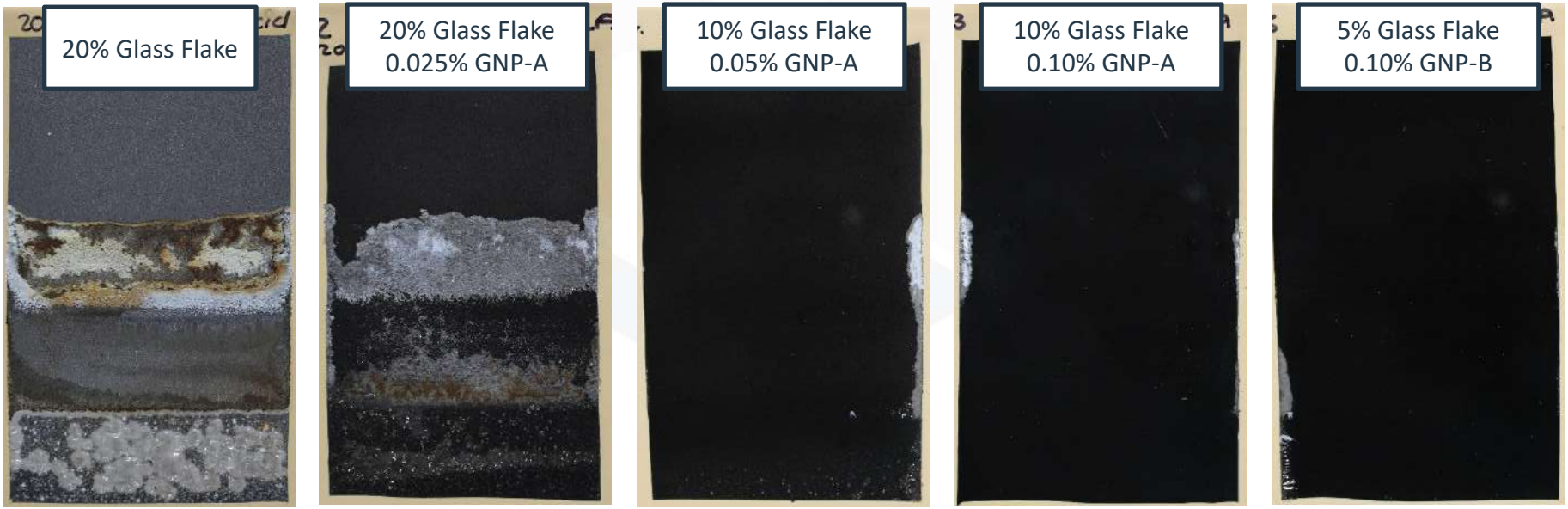


Enhancing Chemical Resistance

10% Lactic Acid Resistance

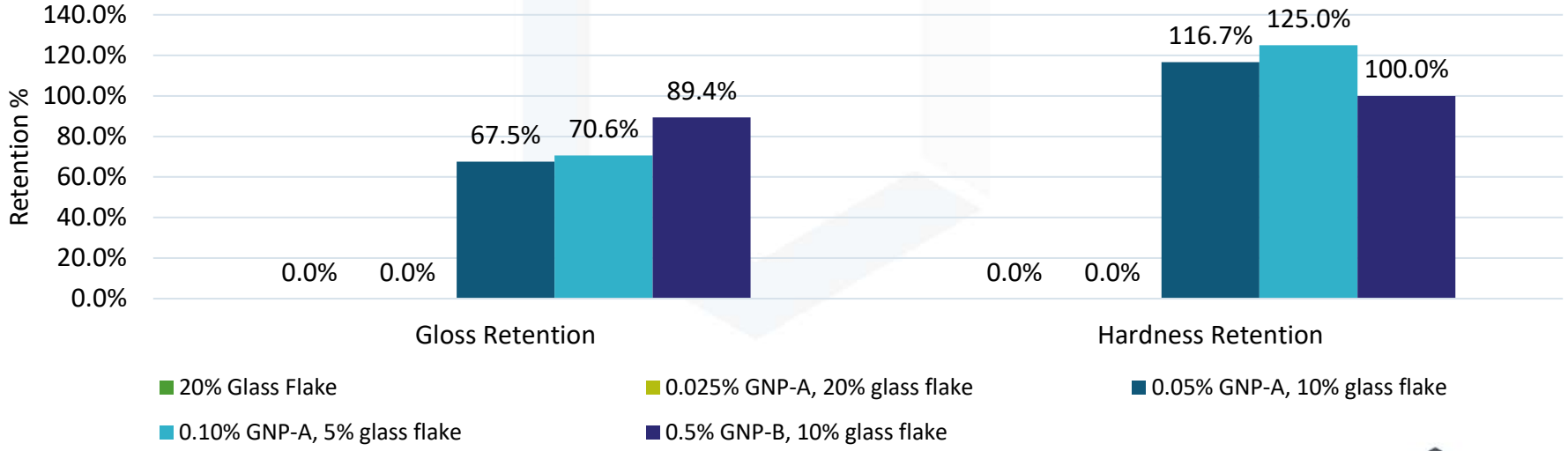
- Glass flake and high glass flake hybrid untested due to failure of the coating

Gloss Retention:

- Retention increased with higher loadings of GNP-A content and decreased glass flake
- GNP-B hybrid shows the highest level of retention

Hardness retention:

- No loss of hardness for tested coatings.



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Enhancing Chemical Resistance

10% Sodium Hypochlorite Resistance

- **Complete failure** of the glass flake coating, rusting in immersed area
- **No visual change** for graphene coatings

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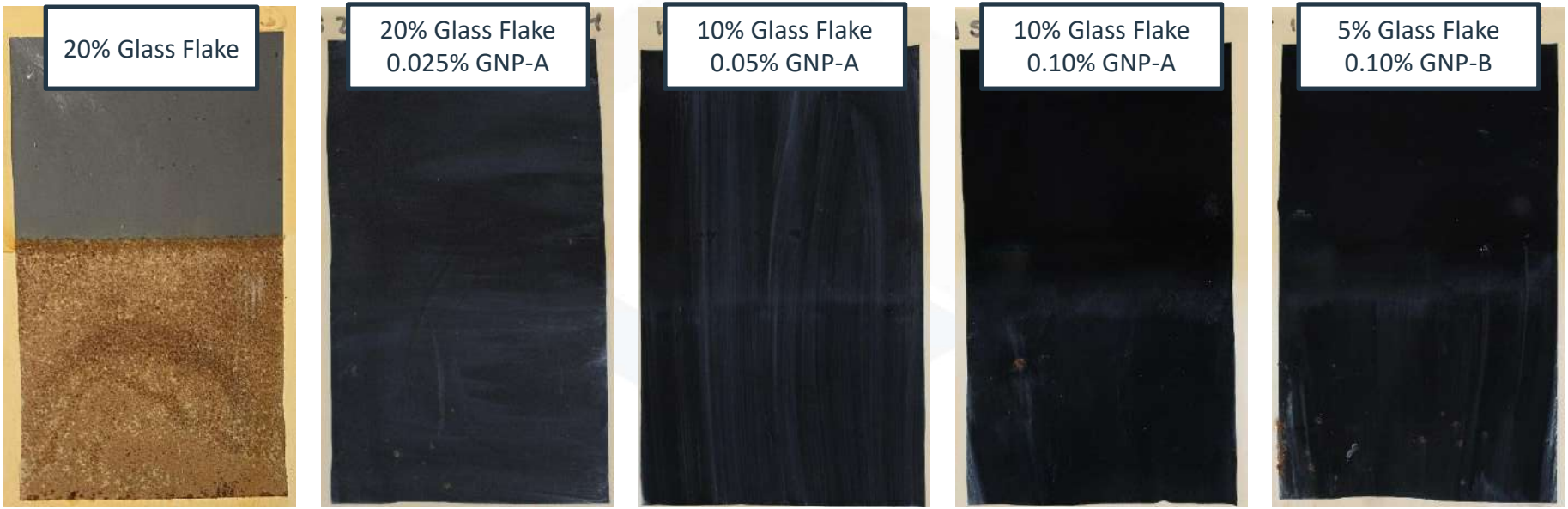

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10% Sodium Hypochlorite Resistance

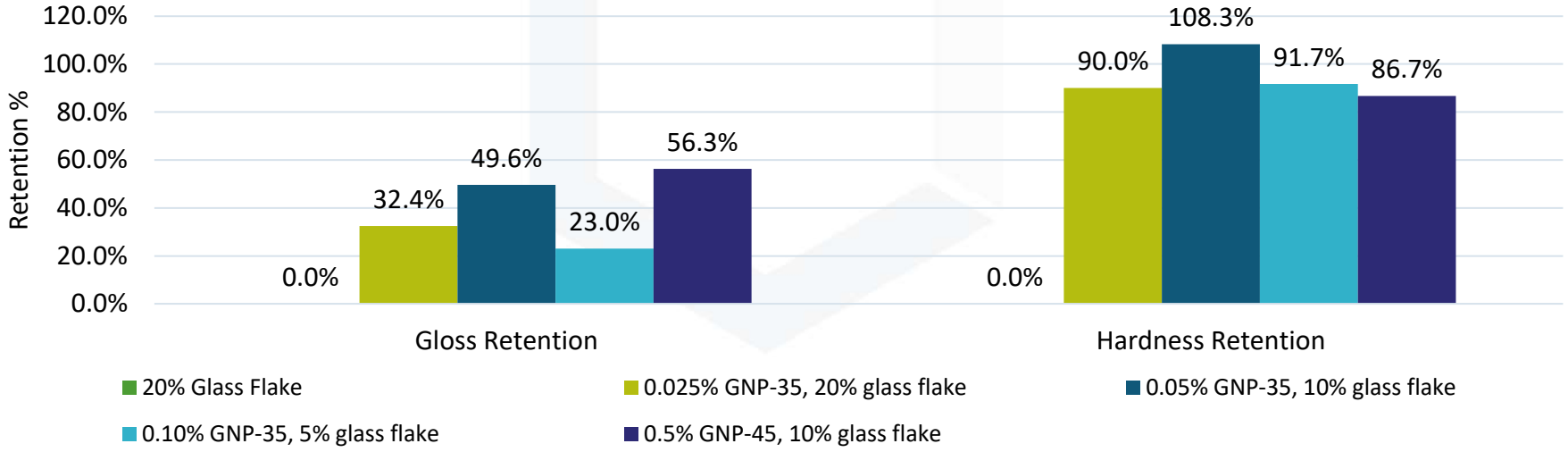
- Glass flake untested due to failure of the coating.

Gloss Retention:

- Better with increasing GNP-A loading
- Highest loading of GNP-A and lowest glass flake hybrid.

Hardness retention:

- Better for mid-level GNP-A hybrid, slightly higher than the baseline sample.



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Discussion

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Discussion

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Glass Flake vs. Graphene

- ***Hardness Retention Properties***
  - Typically glass flake coatings have a higher initial hardness than graphene coatings
  - **Improved retention of hardness** under the exposure media is better for **graphene-enhanced coatings**
  - **Low loading** of graphene required to achieve a **significant performance increase**
- ***Gloss Retention Properties***
  - Graphene coatings record **higher levels** of initial gloss compared to glass flake coatings
  - Retention of gloss is typically **improved** with graphene-enhanced coatings
- ***Visual Assessment Properties***
  - Graphene coatings are **less susceptible** to blistering and other visual indicators of failure
  - Multiple **glass flake coatings completely failed**, others rusted significantly

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Discussion

Glass Flake & Graphene Hybrids

- ***General Observations***
  - Combines the initial **hardness of glass flake** with the retention and **enhanced barrier properties** of graphene
  - High aspect ratio of graphene is advantageous
- ***Hardness Retention Properties***
  - Hardness of glass flake and graphene hybrids is higher than graphene coatings
  - 4H pencil vs. H/F pencil of graphene coatings
- ***Gloss Retention Properties***
  - Gloss retention of hybrid coatings is higher than pure glass flake coatings and pure graphene coatings in some cases

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Conclusions

- Graphene-enhanced coatings can offer significant potential performance advantages compared to traditional glass flake coatings across a range of immersion media
  - ✓ **Gloss** – higher initial values and better retention
  - ✓ **Hardness retention** – Better retention of initial hardness throughout the exposure
  - ✓ **Blistering** – Significantly improve resistance to blistering
- Graphene/Glass flake hybrid systems show promising synergistic effects
  - ✓ **Increased hardness** initial hardness from glass flake
  - ✓ **Superior barrier** from graphene nanoplatelets
- Graphene coatings can offer a **robust** and effective way of creating **protective barrier coatings**
- Graphene offers **improved retention** of physical properties and **resistance to chemical immersion**
- Future work to develop fully formulated coatings to demonstrate industrial utilisation of graphene in industrial coatings

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