

PROJECT REF. NBI-13-0010
August 2017

PAINT ABSORPTION ANALYSIS

- Increase workplace safety
- Create less waste
- Lower the costs of spills and leaks



SpillFix®

Environmentally responsible solutions for a safer workplace



American Green Ventures (US), Inc.
180 Towerview Court Cary
North Carolina 27513
www.spillfix.com

INTRODUCTION

The use of hydrocarbon solvents for cleaning utensils and accident sites in the paint and coatings industry represents a real threat to ground water and water supply/ treatment facilities throughout the United States and developing countries around the world. In truth too much paint is illegally poured down the drain every day. SpillFix presents not only an environmentally responsible alternative to (wet) clean up, but a more economical alternative.

The following finding conclude that SpillFix (made from coconut husks) is a more affective cleaning solution for paint and coatings than any solvent or procedure. American Green Ventures, a subsidiary of the Galuku Group Ltd, is a leading supplier of coconut husks (coir-cocopeat) and has an extensive range of products for the horticulture, hydroponics, bio-films, chemicals and the pet litter industry.

The Galuku Group has investigated the scientific rationale and understanding behind one of their key products (SpillFix) in the chemicals industry. Spillfix is a chemical free, all-natural universal organic absorbent used for spills including oils, fuels, solvents and chemicals. Spillfix is also an excellent absorbent for paints. Preliminary studies have indicated that when Spillfix comes in contact with wet paint (for example in a paint can), Spillfix quickly absorbs the paint leaving the storage container clean and paint-free.

Paints contain various volatile compounds, chemicals, and other hazardous compounds that can cause serious and harmful effects to the environment when disposed inappropriately. Countries such as the US and Australia have strict laws against paint disposal due to its hazardous nature. The ability of Spillfix to absorb paint from paint containers can have a huge market potential in such countries.

For this reason Galuku P/L has requested a scientific study on understanding the mechanisms behind Spillfix's ability to absorb paint. Paints are essentially composed of a binder, pigment and solvent. The pigment gives the paint its color and finish. In addition, pigments also assist in corrosion and weathering. Pigments can either be organic and inorganic. Binders are used in paint to "bind" the pigment to the surface. They are usually polymers and play no other functional role.

Solvents make up the bulk of the paint and act to solubilise the binder. For emulsion paints, the solvent is simply water, while for resin-based paints, the most commonly used solvent is mineral turpentine.

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RESULTS & DISCUSSION

Solvent Absorption

The aim of this experiment was to examine the solvent absorption and retention capacity of coconut coir.

Methodology

Three different sizes of coconut coir were explored for the solvent absorption study; course, Spillfix and fine grade coir. 5, 10 and 20 grams of each grade were added to an air tight container. To this was added a particular paint solvent. The resulting mixture was then stirred (400 rpm) for 5 mins and closed. The solution was left to swell overnight.

The resulting product was then gravity filtered until all solvents were removed. The wet coir was then measured (wet weight) and compared against the three grades of coir. A table of solvents used for this experiment is outlined below. These solvents were chosen as they are the most commonly used solvents in the paint industry.

Table: 1.

	Solvent Name
Solvents used in coir absorption	Water
	Ethanol
	Isopropanol
	Mineral Turpentine (Mineral Turp)
	Acetone



RESULTS & DISCUSSION cont.

Table 2 Indicates the wet weight of material following swelling and absorption overnight.

It can be easily concluded that, of the 5 most commonly used solvents in paint, water is most readily absorbed by coconut coir followed by alcohols and non-polar solvents. This is due to the lignocellulosic fibers in the coir material.

The lignocellulosic fibers in coir are hydrophilic. They contain large amounts of hydroxyl groups on the cell walls of plant fibers. When water comes into contact with these groups during the absorption study, the hydroxyl groups form hydrogen bonds with the water molecules holding them in place and becoming hydrated and moist. This hydrogen bonding attraction between the water molecules and coir is the reason why coir absorbs more water than any other solvents in Table 1.

In addition, ethanol and isopropanol also contains functional groups that can participate in hydrogen bond formation however the hydrogen bonds that these alcohols form are significantly weaker. This is because water molecules contain twice as much hydrogen bonding than isopropanol and ethanol. Mineral turpentine and acetone contain no functional groups that can contribute to hydrogen bonding and therefore is expected to be least absorbed and retained by the coir fibers. The results in Table 1 therefore are expected.

Table: 2

All weights are in (g)	Dry weight of coir	Water (wet weight)	Ethanol (wet weight)	Isopropanol (wet weight)	Mineral Turpentine (wet weight)	Acetone (wet weight)
Fine Grade Coir	5	42.89	32.84	23.42	21.19	19.61
	10	77.26	55.43	51.74	51.44	48.62
	20	183.25	122.6	107.53	109.43	110.14
Coarse Grade Coir	5	21.88	17.27	24.67	28.36	24.66
	10	67.47	55.4	46.49	46.87	52.39
	20	130.42	106.31	99.81	99.51	87.50
SpillFix	5	32.73	21.99	23.92	24.25	23.81
	10	69.96	50.72	47.98	52.87	39.77
	20	127.28	91.85	90.77	87.71	76.61

ABSORPTION CAPACITY

Figure 1-3 indicates the absorption capacity of Spillfix, course and fine grade coir in the presence of the 5 commonly used solvents in paint.

Figure 1 – Solvent absorption by Fine Grade Coir

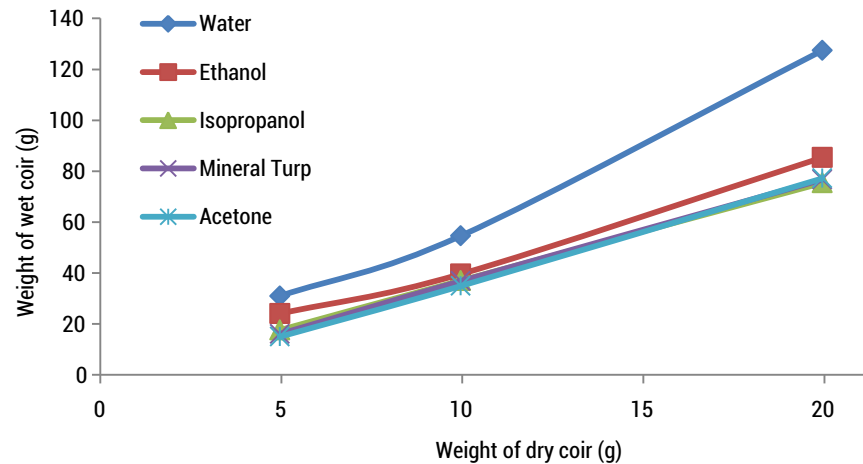


Figure 1-3 represent the total weight of solvent held by the 3 types of coir. It is expected that the percentage of water absorbed is irrespective of the dry weight of coir and therefore a linear relationship would exist between the coir and its absorption capacity. Figure 4 (following page) shows these relationships.

Figure 2 – Solvent absorption by Course Grade Coir

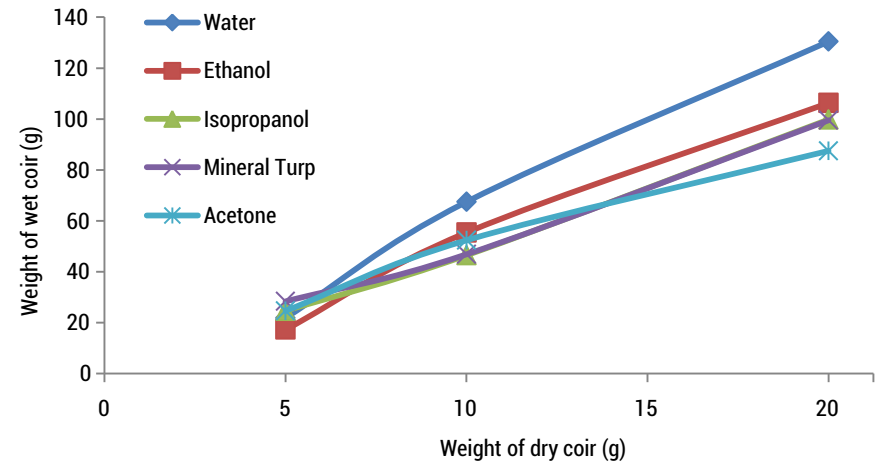
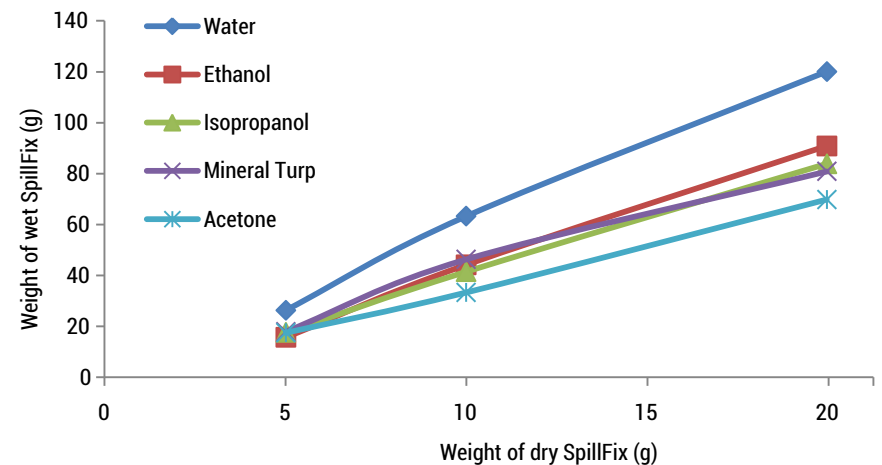


Figure 3 – Solvent absorption by SpillFix



COMPARATIVE ABSORPTION

Figure 4-8 represents the absorption capacity of the three different grades of coir in the presence of different solvents. It is noticeable that the absorption capacity is dependent on the size of the coir particles. The smaller the coir, the larger the surface area of the fibers and this results in greater absorption capacity.

In addition, decreasing the coir size also decrease the number of air-filled pore spaces and increasing the holding capacity of coir. It is therefore logical to assume then that smaller sized particles will have a higher solvent retention capacity. This is evident thought the 5 solvents examined. Generally, the fine grade coir has a much higher absorption capacity than Spillfix and course grade coir. This becomes more evident with increasing weight of the dry coir.

It can also be noticed that with water, mineral turpentine and acetone, the absorption capacity remains constant regardless of the dry weight of coir used. For example in the case of water, an 800% absorption is achieved by fine grade coir regardless of

the amount of coir used. In comparison, both course grade coir and Spillfix have an absorption capacity of approximately 600%. It is also worthwhile to note from these experiments that the minimum absorption capacity for coconut coir is above 300%.

Figure: 4 – Percentage Absorption of Water

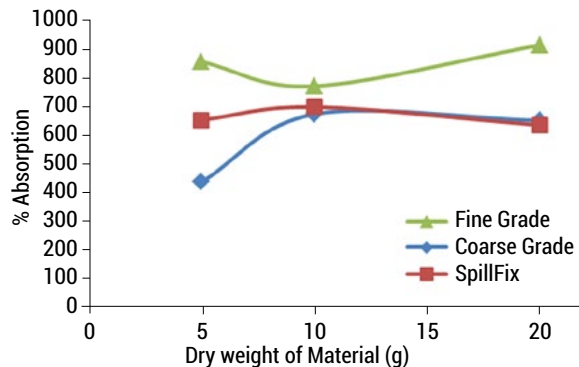


Figure: 7 – Percentage Absorption of Mineral Turp

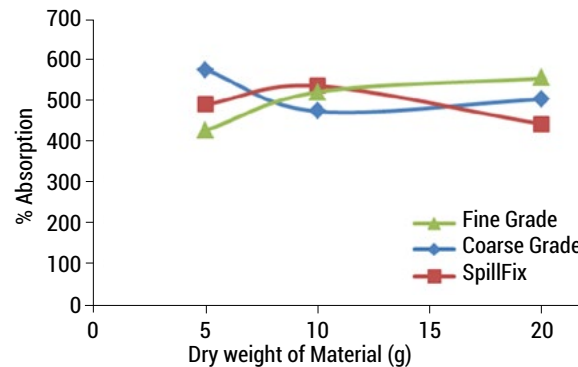


Figure: 8 – Percentage Absorption of Acetone

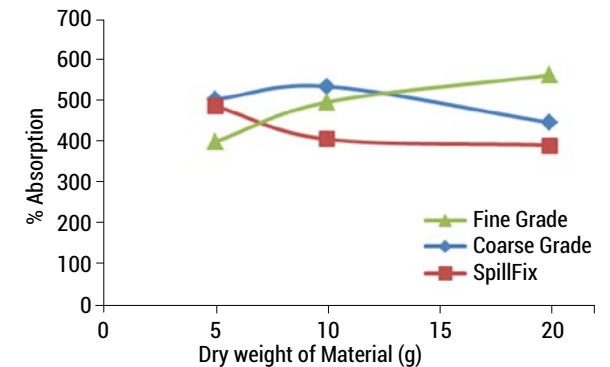


Figure: 5 – Percentage Absorption of Ethanol

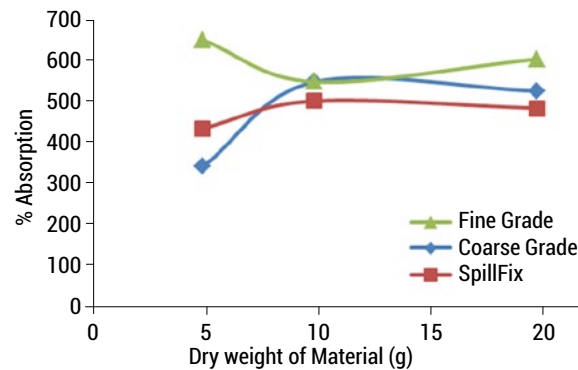
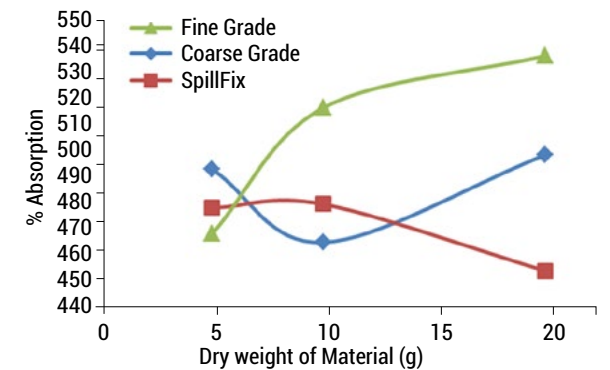


Figure: 6 – Percentage Absorption of Isopropanol



CONCLUSION – Solvent Absorption

Of the three sizes of coir used in the above experiments, fine grade coir has the highest solvent absorbing capacity followed by Spillfix and course grade coir for any given solvent. In addition, the highest absorbing solvent was water followed by ethanol and the remaining three volatile solvents.

These results indicate that the fine grade solvent would be the suitable coir of choice given its higher solvent absorption capacity. Moreover, the results above suggest that coconut coir would be most suitable for paints that contain water as a solvent. However, it is worthwhile to note that following the absorption experimentation, the coir was left to sit openly at room temperature to examine the solvent retention capacity.

Results indicate that when left in the open, the solvents slowly evaporates into the atmosphere. Visual observation of the coir indicates drying out of all 5 solvents. The retention capacity was inversely proportional to how volatile the solvent was. For example, mineral turpentine and acetone evaporated within 10 mins of exposure to the atmosphere while water took over a day to evaporate. These results suggest that there is no chemical or ionic interaction of the solvents towards coir. It is simply the swelling of the coir to retain the solvent. The hydrogen bonding present in solvents such as water and alcohols are only weak interactions with the coir. Thus, water, ethanol and isopropanol eventually evaporated.



DYE ABSORPTION

Of the three sizes of coir used in the above experiments, fine grade coir has the highest solvent absorbing capacity followed by Spillfix and course grade coir for any given solvent. In addition, the highest absorbing solvent was water followed by ethanol and the remaining three volatile solvents.

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The aim of this experiment was to test the absorption of various paint dyes by coir. These dyes were found to be commonly used in the paint industry. 4 dyes were chosen namely; yellow, green, blue and red to cover the visual spectrum. Described below are the methods used for their analysis.

METHODOLOGY

Azo (Yellow) dye

Azo yellow (3.29 mg) was carefully weighed out and added to a 100 ml volumetric flask. To this was added 100 mls of chloroform and shaken until dissolved. 20 mls of the dissolved solution was accurately measured out and made into 100 mls using chloroform. The resulting solution was then added to an air tight glass container and to this was added Spillfix (5 g). The final mixture was closed, shaken and UV-vis measurements were taken over a period of 24 hrs to observe the absorption capacity of Spillfix.

Phthalocyanine Blue

Phthalocyanine blue (3.11 mg) was carefully weighed out and added to a 100 ml volumetric flask. The dye was then made up to 100 mls with chloroform. The solution was then shaken and sonicated until it was dissolved. The solution was added to an air-tight glass flask and to this was added Spillfix (5g). The resulting mixture was sealed air-tight and shaken for a few minutes. UV measurements were taken at various time intervals over a period of 24 hours.

Phthalocyanine Green

A similar experiment to Azo (Yellow) dye was performed with phthalocyanine green (4.19 mg) in chloroform (100 mls) with SpillFix (5 g).

Red 170

A similar experiment to Phthalocyanine blue was performed with Red 170. Red 170 (2.45 mg) was made up to 100 mls with chloroform and dissolved via sonication. The solution was transferred to an air-tight container and to this was added SpillFix (5 g). The container was closed and shaken for a few minutes. UV measurements were taken at various time intervals over a period of 24 hours.

RESULTS & DISCUSSION

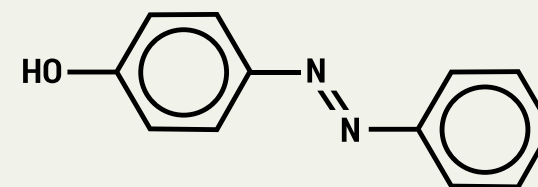
The absorption of the dyes by SpillFix was determined using UV-Vis spectroscopy.

Figure 9 shows the absorption of the Azo (yellow) dye by Spillfix over a period of 24 hrs. The peak identified at approximately 430 nm is indicative of the Azo dye in the absence of the coir. Following addition of the coir, there is no significant change around this region.

This indicates that there is no absorption or adsorption of the Azo (yellow) dye by SpillFix over the 24 hour period. These results are further confirmed by

photographic pictures of the dye in the presence of the material. The results found are expected and can be explained based on the structural nature of the coir. Coir is made up of cellulose, hemicellulose and extractives such as pectins and tannins. These constituents contain numerous hydroxyl and carboxyl groups which allow the coir to be very good absorbers of cations and metals such as sodium, potassium, calcium and magnesium. The yellow dye used in this experiment however contains no metals or cations. This would then explain why the yellow dye was not taken up by the coir over the period of 24 hours.

Figure: 10 – A common azo yellow dye containing two aromatic rings



There is also a small presence of a peak at 680 nm following the addition of SpillFix. This could be due to a discharge of compounds and constituents from the coir material.

Figure: 9 – Color Absorption Capacity of SpillFix

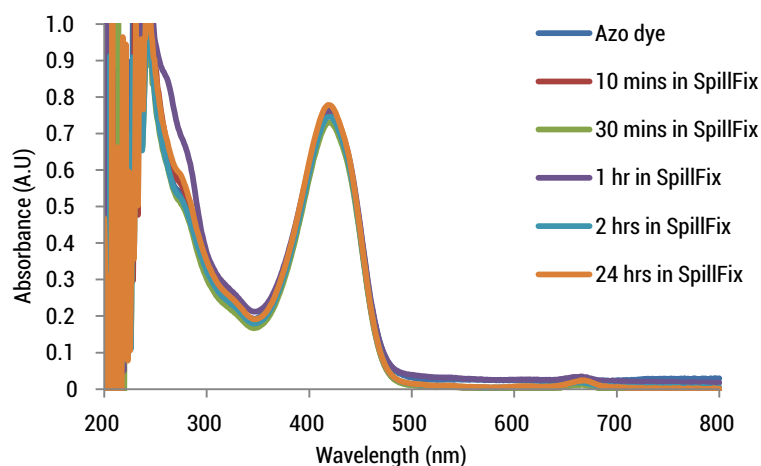
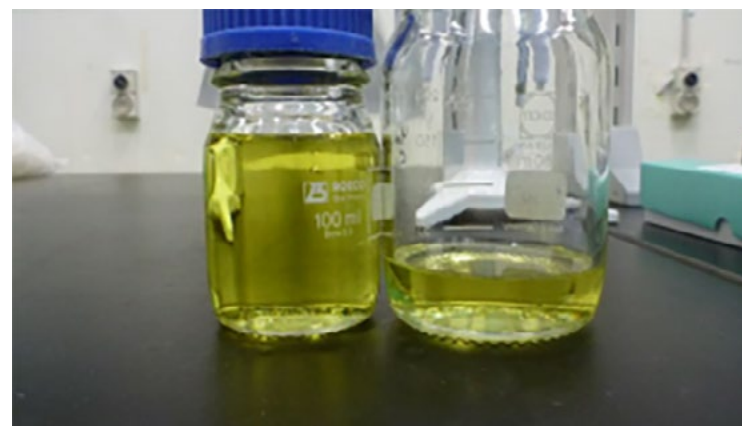


Figure: 11 – Yellow dye before (left) and after (right) filtration



RESULTS & DISCUSSION cont.

Figure 12 represents the UV measurements of phthalocyanine blue in the presence of SpillFix over a 24 hour period.

The line represented in blue is the phthalocyanine blue dye in the absence of SpillFix. The peak present at approximately 680 nm as well as the region of 550-800 nm is indicative of the blue color of the dye molecule.

Upon addition of SpillFix, there is a significant decrease in this region (550-800 nm). These results show that the blue dye is being absorbed by the SpillFix upon first contact. Within the first 10 mins, the 550-800 nm region has completely diminished. This is expected as the blue dye is made of a copper center which when in solution with SpillFix, it would be absorbed.

Figure 13 shows the dye before and after filtration using SpillFix.

Figure: 12 – Absorption of Phthalocyanine blue by SpillFix

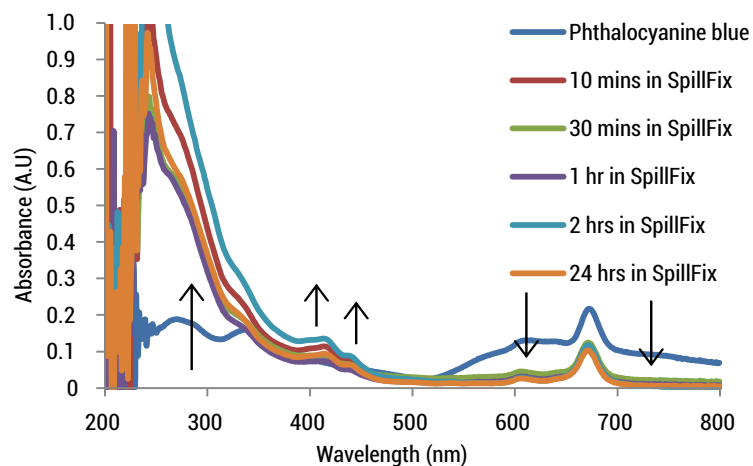
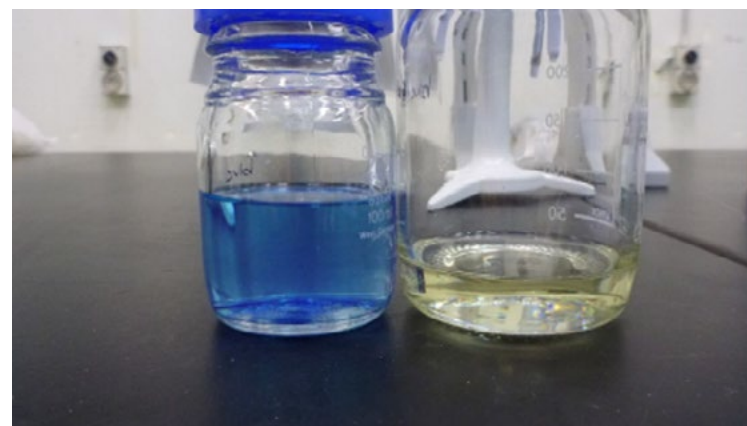


Figure: 13 – Phthalocyanine blue dye before (left) and after (right) filtration.



RESULTS & DISCUSSION cont.

The peak present at 680 nm after the absorption of the blue color is misleading. It is obvious from **Figure 13** that there is no blue color present after the first 10 mins in the presence of SpillFix. This peak shown in **Figure 12** may possibly be introduced from the SpillFix material itself as previously suggested from **Figure 9**.

Figure 12 also indicates the changes in the region 250-450 nm. There is a significant increase in the number of peaks following the addition of SpillFix. This increase could

be a result of materials leaching from the SpillFix. It is widely accepted that coconut fiber contains tannins. When in contact with solvents such as water, the tannin leaches out of the fiber. The UV-Vis spectra of tannins show characteristic peaks around the 300 nm region, similar to those shown here in **Figure 12**. Upon addition of the SpillFix, the SpillFix looks to absorb the metallo blue dye while the tannin seems to leach out of the coir. In addition, these characteristic peaks are further shown in **Figure 14** and **Figure 15**.

Figure: 14 – Phthalocyanine green absorption by SpillFix

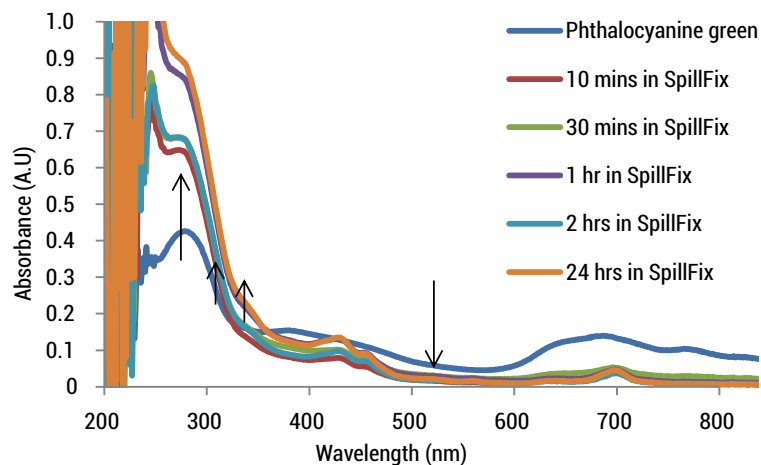


Figure: 15 – Green dye before (left) and after (right) filtration.



Figure: 16 – UV-Vis spectra of red dye absorption by SpillFix.

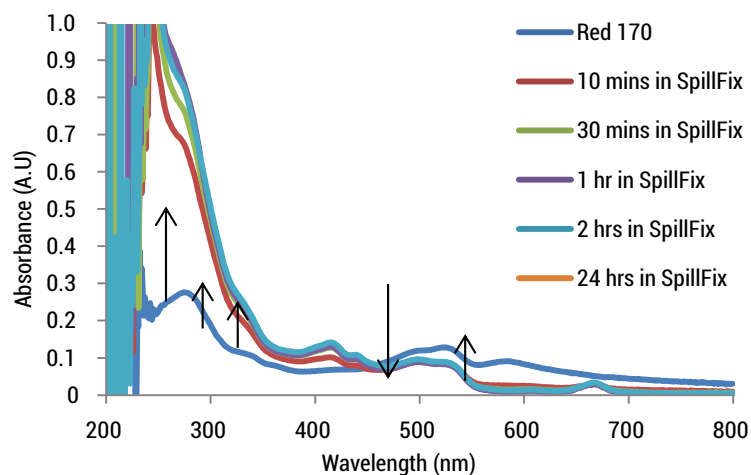
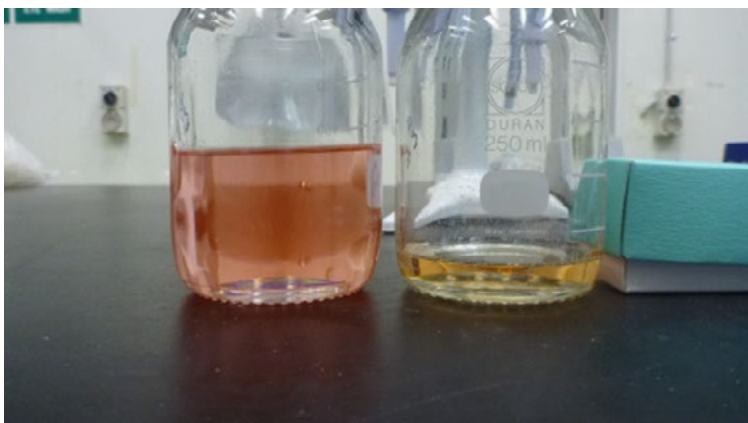


Figure: 17 – Red dye in the absence (left) and presence (right) of SpillFix.



RESULTS & DISCUSSION cont.

Figure 14 - Figure 17 show the UV-Vis spectra of the green and red dyes respectively. Again, there is a characteristic behavior where there is a decrease in signal from 550-800 nm while at the same time, there is a discharge from Spillfix resulting in an increase around the 250-500 nm region. In addition there is a slight yellowing of the solvent following the 24 hour experiment, indicating the presence of tannin. This yellowing is also present in **Figure 13**.

CONCLUSION – Dye Absorption

The study conducted here indicates that SpillFix has a preference for certain types of dyes over others. These dyes show a characteristic nature in that they contain metal ions or net positive charges. Dyes that are organic in nature do not get absorbed by the SpillFix. In addition, insoluble dyes also do not get absorbed by the coir. Furthermore, there is a discharge of the absorbed dyes in the presence of saltwater. This is due to the ability of coir to act as a cation exchanger. I.e, the binding affinity of the coir to dyes are ionic. Ionic binding is much stronger than hydrogen bonding however can be easily broken in the presence of other cations. This was observed when the coir absorbed dye was reintroduced in salty water and shaken. Experiments conducted here elucidated that SpillFix will work really well with dyes that have a net positive charge such as phthalocyanines.

However, due to the weak binding properties of coir, the bound dye can be discharged in the presence of other molecules with positive charge (concentration dependent).

PAINT ABSORPTION

Aim

The aim of this study was to determine the amount of coconut coir required to absorb and remove paint. Two type of paints were investigated namely water and oil based paints. In addition, 3 sizes of coir were used. These were fine grade, course grade coir and SpillFix.

Method

50 grams of paint (water and oil based) was added to a glass beaker and to this was added 5 grams of coir. The coir was then manually mixed in with the paint.

This process was repeated until all paint was absorbed by the coir. **Figure 18** depicts step-by-step process of the coir absorbing paint.

Table 3 (following page) shows the total paint used for the absorption of 50 grams of paint by different particle size coir fibers (Fine grade/Course grade coir and SpillFix).

Figure: 18 – Step-by-step paint absorption process



PAINT ABSORPTION cont.

Table 3 shows that the total amount of course grade coir and Spillfix required to absorb 50 grams of paint is 20 grams. This equates to a 250% absorption capacity. Thus for every 100 grams of paint spill, only 40 grams of Spillfix or course grade coir is required. This is regardless of whether the paint is oil based or water based. The least performing coir was the fine grade. It was noticed that the smaller the particle size of the coir, the harder it was to absorb and remove all the paint. Larger and fibrous coir aid in removing the dried out paint from the source while finer grade particle lack the structural network to remove the dried paint.

Furthermore, it was observable that although the coir absorbed the oil based paint, it took much longer to dry. Samples of the coir that were stored for up to a day was still wet in the presence of the oil based paint. This suggests that the oil based paint requires a much larger amount of coir to absorb the same amount of paint to dry. This could be due to the nature of the paint. Oil based paints generally take much longer to dry than water based paints. Repeat experiments with double the amount of coir showed to dry out the oil based paint at the same rate as the water based paint. It is also important to note the method of absorbing the paint. A two stage process was used.

Table 3 shows the total amount of coir required, but does not discuss the method used to absorb the paint. The process for absorption was a two stage process whereby 5 grams less than the total amount of coir was used to absorb the 50 grams of paint then, once absorbed, 5 grams of fresh coir was added to act as a physical scraper to remove all the paint from the walls of the container.

For example, the total amount of Spillfix used to absorb the paint was 15 grams. To this was added an extra 5 grams to scrape off all remaining paint from the walls of the container. This gave a final amount of 20 grams of Spillfix.

The paint absorbed coir was allowed to dry overnight at room temperature. This was then placed in various concentrations of sulphuric and hydrochloric acid (0.001-1 M) to see if there was a discharge from the coir. Results indicated no such discharge unlike solvent and dye absorption studies.

Table: 3

Name	Type of paint	Amount of paint	Total material required for absorption	% Absorption
Fine Grade	Water based	50g	25g	200
Coarse Grade	Water based	50g	20g	250
SpillFix	Water based	50g	20g	250
<hr/>				
Fine Grade	Oil based	50g	30g	167
Coarse Grade	Oil based	50g	20g	250
SpillFix	Oil based	50g	20g	250

CONCLUSION – Paint Absorption

Although conclusions from dye and solvent absorption studies indicate weak interaction of coir with components of paint, together there is a different type of interaction. With paint, the coir has four functional properties.

1. Absorbing affinity – the coir absorbs the paint.
2. Scraping ability – The rigid structure of the coir allows for the coir to act as a strong scraper to remove any semi hardened or hardened paint off the walls of the container.
3. Catalytic ability – the coir looks to draw out the solvents and moisture from the paint and allowing the resin to dry the paint on the coir at a much quicker rate than alone.
4. Retention – once the paint dries on the coir, there is no discharge of the paint even under acidic conditions.

MICROSCOPY

Aim

The aim of this study was to explore the interaction of the paint with coir at a microscopic level. For simplicity, to this stage of the report, paint-coir interactions have been discussed as “absorbed”. However, there is a distinct difference between absorbed and adsorbed. Microscopy study herein will look to discuss whether the coir-paint interaction is;

1. Absorption
2. Adsorption
3. Both

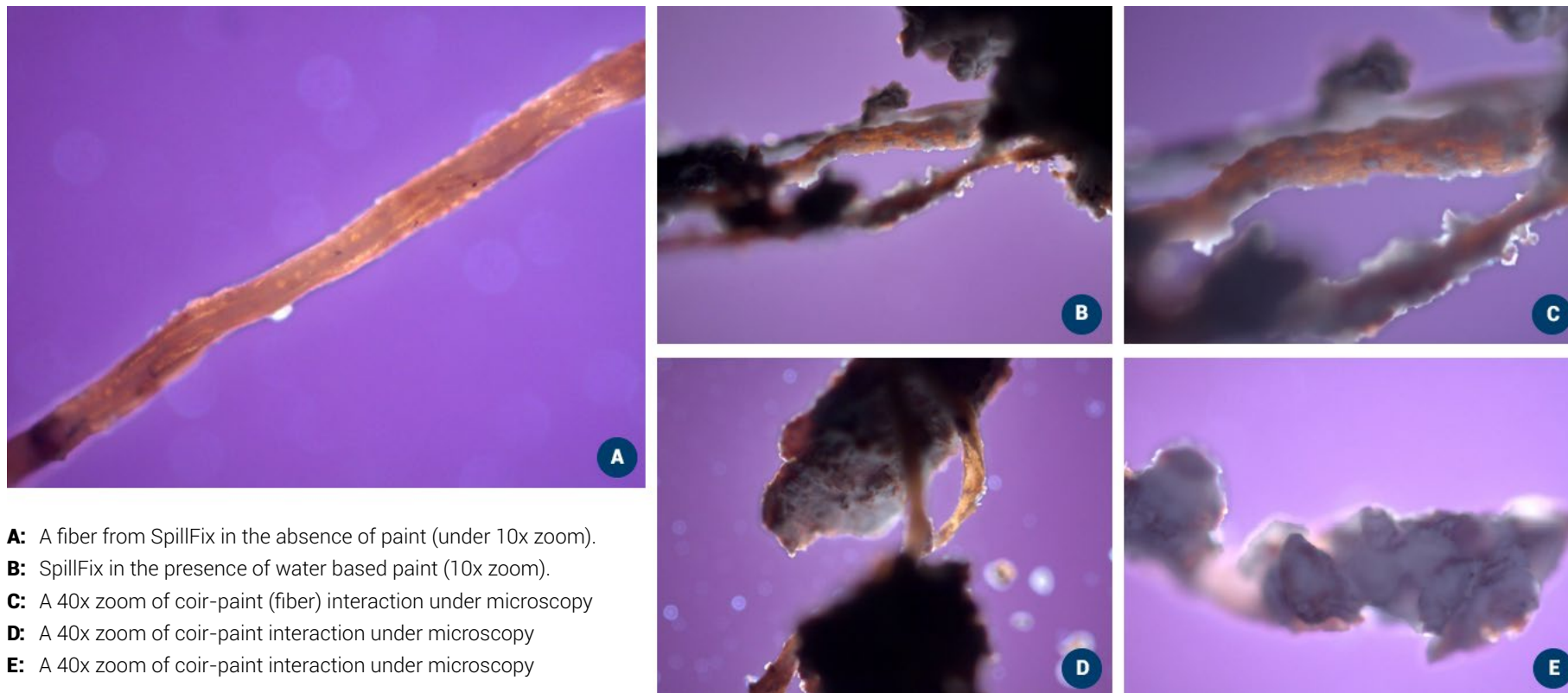
METHODOLOGY

Samples that were absorbed in water based paints were allowed to completely dry. These samples were then analyzed using a Carl Zeiss Axiom microscope with 5x, 10x, 40x and 100x zoom.

RESULTS

Images A – E show paint-coir interaction under microscopy. These observations show that the paint may only be present on the surface of the coir. A closer zoom (40x, Figure 17) of the fibers also shows the same. In addition, non-fibrous coir also shows absorption preferentially. However this does not rule out the possibility

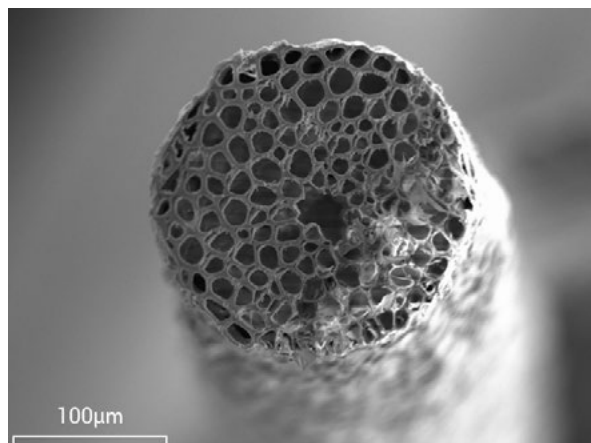
of adsorption. For adsorption studies, energy dispersive X-ray spectroscopy under a scanning electron microscope was utilized. This was achieved with the assistance of The Australian Centre for Microscopy and Microanalysis. A written report was provided by the University of Sydney.



- A:** A fiber from SpillFix in the absence of paint (under 10x zoom).
- B:** SpillFix in the presence of water based paint (10x zoom).
- C:** A 40x zoom of coir-paint (fiber) interaction under microscopy
- D:** A 40x zoom of coir-paint interaction under microscopy
- E:** A 40x zoom of coir-paint interaction under microscopy

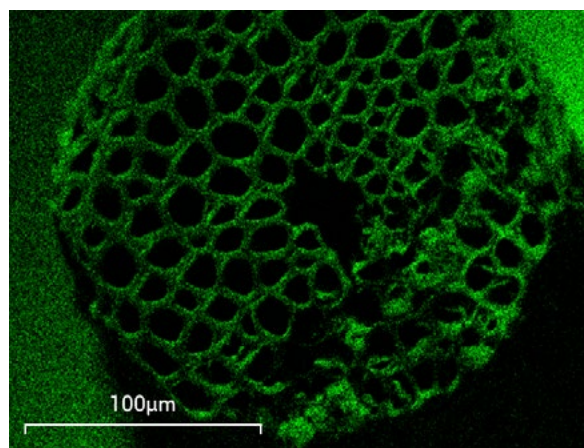
FURTHER MICROSCOPY ANALYSIS

Three samples of SpillFix material, with and without paint were sent to the ACMM for analysis and comparison using microscopy techniques. Samples were labeled as blank, 5gm, 10gm and 20gm. The aim of the analysis was to determine whether paint was taken up into the material or remained on the surface.

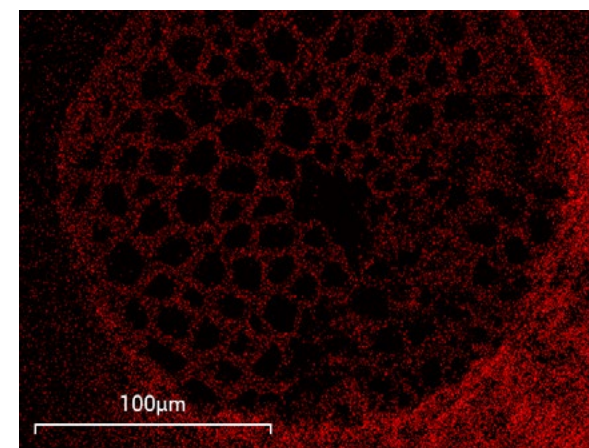


5g SpillFix sample absence of paint

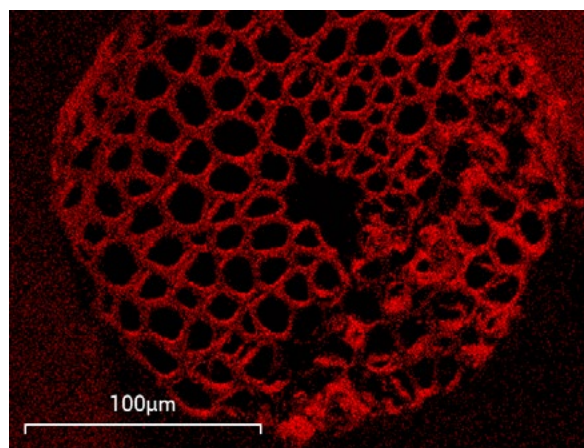
The 5g Sample was prepared by freeze-fracturing and imaged in a scanning electron microscope (SEM) using secondary electrons. Energy dispersive X-ray spectroscopy (EDS) was used to analyze cross sections through the samples to determine the location of the titanium-based paint.



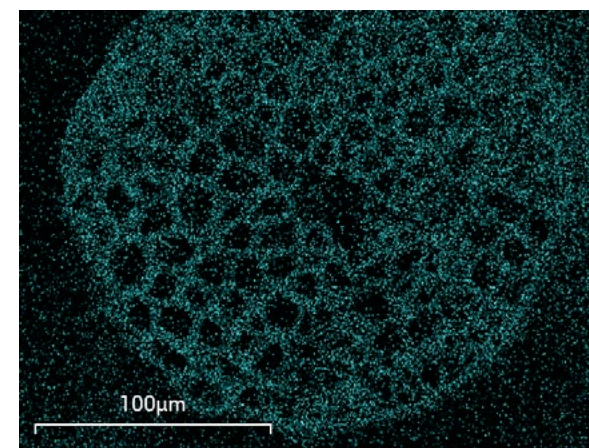
5g SpillFix-paint sample – C K Series



5g SpillFix-paint sample – Al K Series

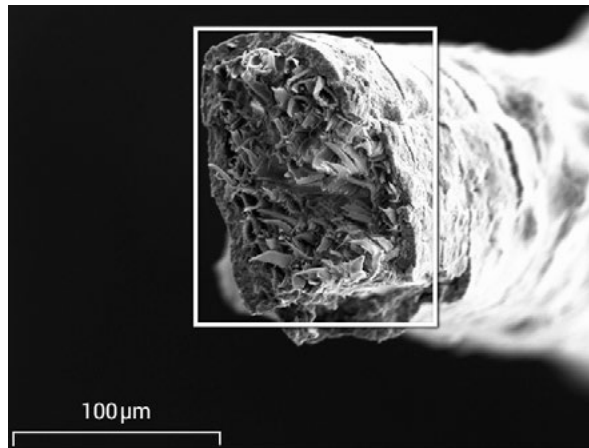


5g SpillFix-paint sample – O K Series

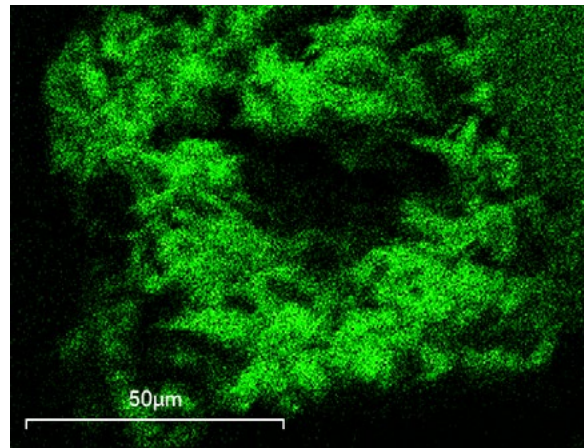


5g SpillFix-paint sample – Cl K Series

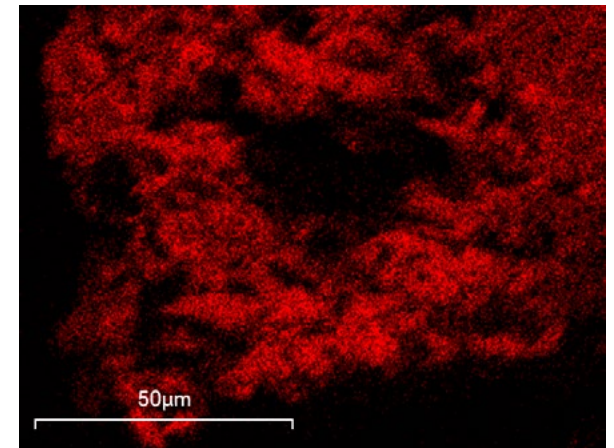
10g SAMPLE MICROSCOPY ANALYSIS



10g SpillFix sample absence of paint

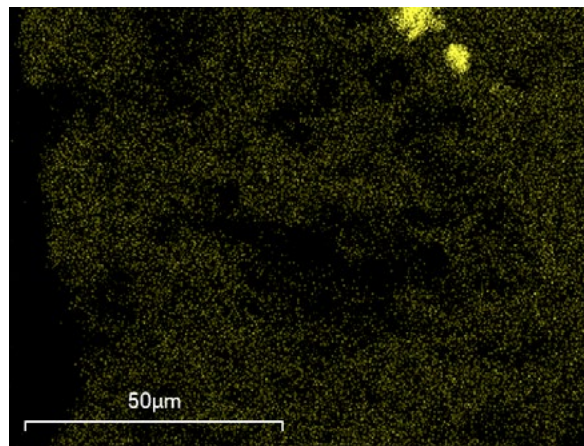


10g SpillFix-paint sample – C K Series

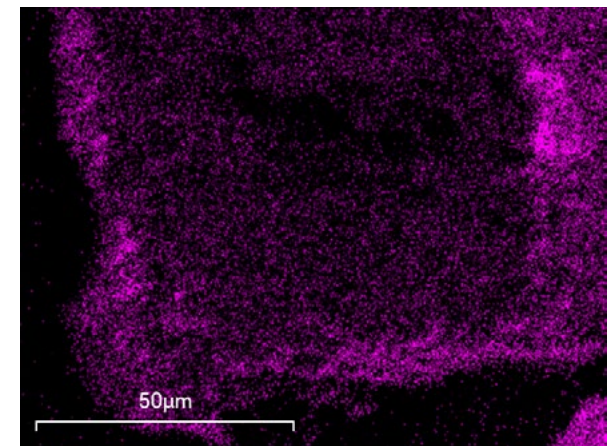


10g SpillFix-paint sample – O K Series

The 10g Sample was prepared by freeze-fracturing and imaged in a scanning electron microscope (SEM) using secondary electrons. Energy dispersive X-ray spectroscopy (EDS) was used to analyze cross sections through the samples to determine the location of the titanium-based paint.

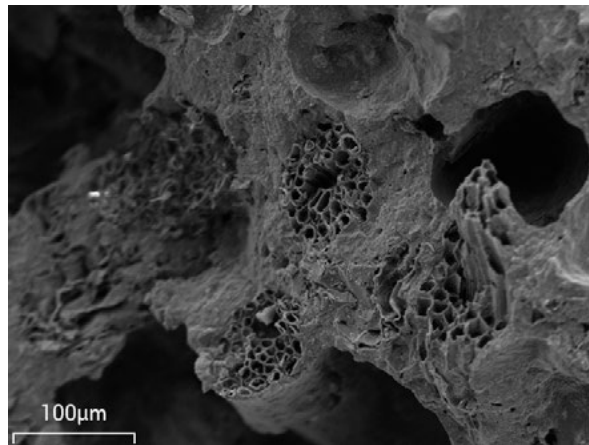


10g SpillFix-paint sample – Na K Series

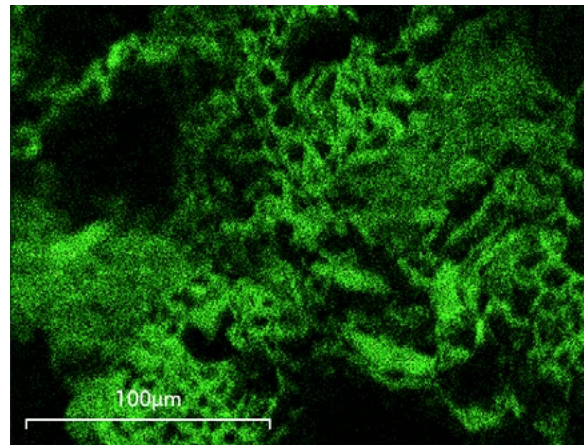


10g SpillFix-paint sample – Al K Series

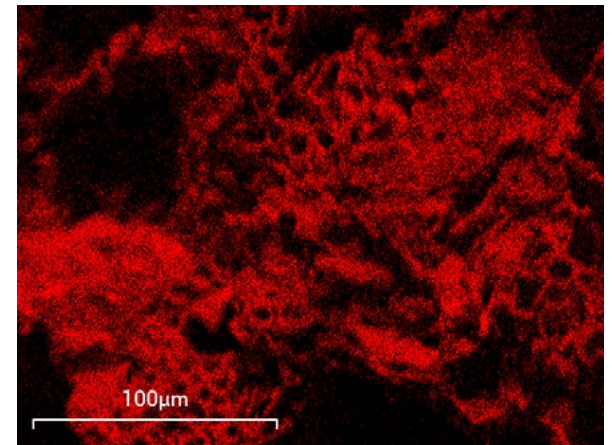
20g SAMPLE MICROSCOPY ANALYSIS



20g SpillFix sample absence of paint

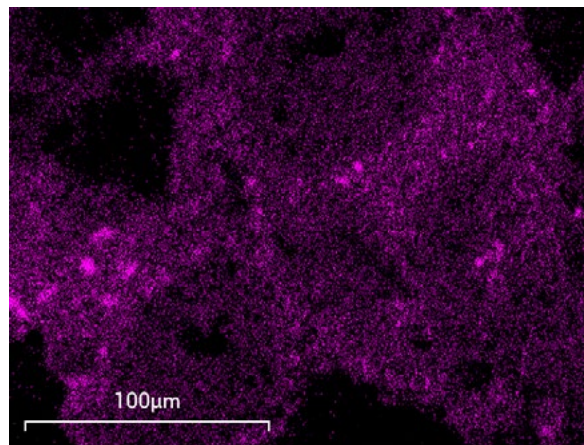


20g SpillFix-paint sample – C K Series

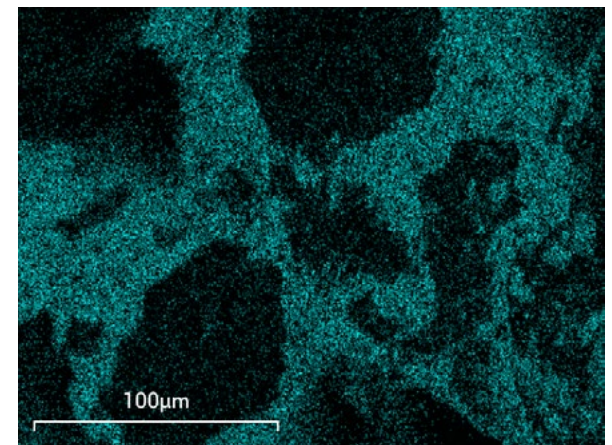


20g SpillFix-paint sample – O K Series

The 20g Sample was prepared by freeze-fracturing and imaged in a scanning electron microscope (SEM) using secondary electrons. Energy dispersive X-ray spectroscopy (EDS) was used to analyze cross sections through the samples to determine the location of the titanium-based paint.



20g SpillFix-paint sample – Si K Series



20g SpillFix-paint sample – Ti K Series

SOURCE

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