

METHOD

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# Microplastic paint particle production for spiking experiments; silicone rubber as application material provide high yield with low effort

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## Abstract

Paint particles are a highly-important but as-yet overlooked type of microplastic commonly found in coastal sediment, although research interest is growing. There is a need for paint particles as laboratory standards for spiking experiments, however, given paint is generally only available for purchase in liquid form, a new method is needed to reliably produce laboratory-grade paint particles. The main distinguishing issue with paint particle production (as opposed to other microplastic production) is the paint must be applied to a surface in layers before then being decoupled to be processed into particles. Since paints, and especially primers, are designed specifically to strongly adhere to surfaces, using the right application material is highly important. This study tests the time and material efficiency of 2 different application materials; laboratory wax film (i.e. Parafilm®) and silicone rubber sheets (i.e. silicone baking sheets). Silicone rubber was on average 36 × more time-efficient than laboratory wax and was also 8 × more material-efficient (8 × more paint particles were produced per liquid paint used). Indeed, silicone rubber provided an essentially-perfect decoupling process, as the paint could be easily peeled away in a solid and complete layer. As such, for the future production of paint particles for laboratory purposes, silicone rubber is highly recommended as an application material.

**Keywords** Paint particles, Microplastics, Laboratory standards, Silicone rubber

## Introduction

The study of microplastic pollution is multi-faceted. While environmental sampling and monitoring make up the bulk of work on the topic, laboratory trials are an essential part of microplastic research, especially toxicological impacts. In order to perform such studies, microplastic standards are needed. For conventional

microplastics (typically polyethylene, polypropylene, polyvinyl chloride, polyethylene terephthalate etc.), methods are already established for production of standards [1], although microplastic standards can also be ordered from polymer production companies, and even these days from scientific suppliers such as Sigma-Aldrich (Missouri, USA) [2]. However, for more unusual particulates this is not always the case. In 2016, Cole [3] published a method for the production of microplastic fibres for spiking experiments, since microplastic-fibres are not typically commercially available yet a highly important environmental MP, being found in virtually all samples globally [4]. As was the case pre-2016 for microplastic

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fibres, for the study of microplastic-paint particles there is no established production methods or suppliers.

Paint particles, typically found as flakes within the microplastic size range (<50 µm), are very prevalent in many coastal sediments [5]. Indeed, in areas where ship activity is high, such as harbours and marinas, they are often the most abundant type of microplastic found in sediment [5, 6], and can be numerous enough to make up an appreciable contribution to total sediment mass. For example, an estimated 1% of sediment mass was deemed to be paint particles in a British tidal inlet [7], while in a Brazilian harbour paint particles made up as much as 4.4% total sediment mass [8]. However, the study of paint particles, especially when considering the overall volume of microplastic research, is fairly minimal [5]. There are a number of potential reasons why this might be the case. Firstly, paint particles are chemically far more complex than conventional plastics, making them challenging to analyse and categorise. This is because, while the binder is polymer-based and can be categorised in a similar way to conventional microplastics, paints even of an identical polymer can still vary greatly since this polymeric content makes up, comparatively to conventional plastics, much less of the total content. So while determining the polymer of a paint particle using typical chemical analysis approaches for microplastic work (such as by FT-IR spectroscopy) is possible, additional approaches to determine the pigments and additives, especially metallic additives, are needed to properly categorise paint particles chemically. Additionally, paint flakes are usually much more brittle than even very weathered microplastics, since the polymeric binder which holds the material together makes up far less of the content. So mechanical mixing or homogenisation steps as part of the methods to process samples before they can be analysed may well cause fragmentation of paint particles, potentially biasing the results, since fragmentation would arbitrarily increase the total particle count. These issues with the methods involved in reliably sampling and identifying paint particles lead to them often being overlooked during toxicological laboratory trial studies. However, paint

particles, due to the high levels of additives, especially some which are specifically designed to inhibit microbiological growth, may have a considerable influence on the environment. Therefore, interest in paint particles is growing and as such the need for standards for spiking trials is needed.

In order to produce paint particles for research trials, paint must be first applied to a surface and dried to remove the solvent, then particles can be produced from this solid layer. To be a good proxy for environmental paints, this should be done in a similar way to how they would be applied naturally, and this typically involves the initial application of a primer base layer before multiple top coats. However, unlike a typical application, to produce paint particles the paint then needs to be removed from the surface which it was applied to so that only the paint is taken forward for future experiments. This paper outlines 2 different methods to produce paint particles for laboratory spiking trials using different application surfaces: laboratory wax film and silicone rubber.

## Materials and methods

4 different paints were selected for testing (2 different primers and 2 different top coats). All paints selected were specifically designed for use in a maritime environment (all marketed as boat paints). Manufacturer details are shown in Table 1. Primer 1 was paired with top coat 1 and primer 2 was paired with top coat 2 to give 2 different combinations henceforth referred to as P1 and P2.

Laboratory wax film (Parafilm<sup>®</sup>, Sigma- Aldrich, Missouri, USA) and silicone rubber sheets (Koksi Corp. Ltd, UK) were used as application material. Paint was applied by brush in even layers. In all cases 2 layers of primer were applied followed by 3 layers of top coat. All layers were left to dry for a minimum of 10 minutes before application of the next layer. Painting and drying was conducted outdoor in a covered area. Temperature varied from 7 to 15 °C during the drying period.

Methods to separate paint from the application material varied based on what was possible. After 24 h the paint had partially dried but was still tacky to the touch and flexible. At this point in the drying process, the silicone rubber sheeting could successfully be peeled away from the paint cleanly. 90% of the paint film was peeled away in a single sheet from the silicone, with the remaining 10% left to adhere to allow the paint film to hang from the silicone in free air to expedite drying. The same was not possible for the laboratory wax film and so the paint was left to fully harden on the film. In both cases, the paint took a further 14 days to fully harden.

To separate the hardened layer of paint from the wax film, the film was slowly stretched. This caused the hardened layer to crack into large fragments. A supporting finger was placed behind each fragment and the

**Table 1** Details of paints used for paint particle production

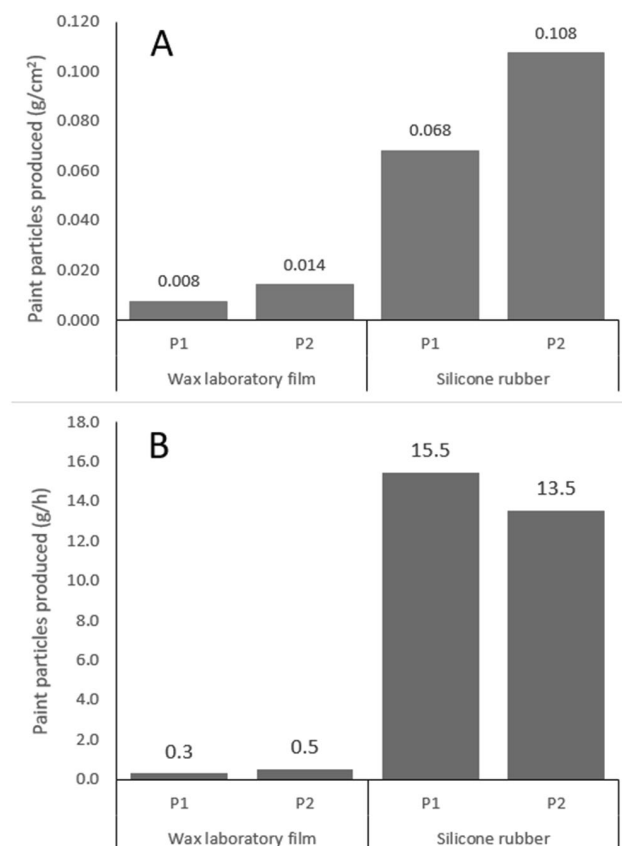
ID	Paint type	Commercial Name	Colour	Manufacturer
P1	Primer	Yachtcare Anti-fouling Primer	n/a	Vosschemie GmbH (Uetersen, Germany)
	Top-coat	Antifouling LB25	Black	Wohlert-Lackfabrik GmbH (Hatten, Germany)
P2	Primer	International Primocon	n/a	Akzo Nobel N.V. (Amsterdam, Netherlands)
	Top-coat	International Ultra 300 Antifouling	Blue	Akzo Nobel N.V. (Amsterdam, Netherlands)

film stretch further until the fragments were released. In many cases this was not possible and the film broke before the fragment was released.

To produce paint particles, hardened paint flakes were placed on a stainless steel sieve stack. For the application of the author, paint particles of 1 mm – 500  $\mu\text{m}$  were required for spiking experiments so a sieve stack was constructed of 2 different mesh sizes: 1 mm, 500  $\mu\text{m}$  and a final collection plate. A stainless steel spoon could then be used to gently crack and agitate the larger flakes on the 1 mm sieve into smaller fragments until they passed the first sieve and collected on the second. These were then poured onto aluminium foil and transferred to glass airtight tubes for storage until required for further experimentation.

## Results and discussion

Paint particle production using silicone rubber sheets as application material was far more efficient than laboratory wax film, both in terms of material-efficiency (mass of paint particles per unit painted area) and time-efficiency (mass of paint particles per unit time). On average, 0.011  $\text{g cm}^{-1}$  was produced using the laboratory wax film



**Fig. 1** Paint production efficiencies. **A:** Material efficiency (amount of paint particles produced at the end of the process as a function of the area of painted surface used at the start); **B:** Time-efficiency (active time spent by user in process steps, not including drying time)

approach while 0.088  $\text{g cm}^{-1}$  was produced using silicone rubber (see Fig. 1A). However, time-efficiency saw the greater difference with an average 36-fold increase in paint particles produced per unit time invested by the researcher (not including drying time). An average of 0.398  $\text{g h}^{-1}$  were produced using laboratory wax while use of silicone rubber enabled an average production of 14.491  $\text{g h}^{-1}$  (see Fig. 1B).

The advantage of using silicone rubber sheets over laboratory wax film is clear based on this data alone, however there are also additional aspects to consider which further recommend the use of silicone rubber. The main issue which hindered the wax film approach was that the film would often tear before fragments of paint could be decoupled. This could be influenced by temperature, since despite a claimed usability temperature range of  $-45\text{ }^{\circ}\text{C} - 50\text{ }^{\circ}\text{C}$  [9], the ability to stretch the film (necessary for detaching paint layers) varies considerably at different temperatures. Based on working observations, it becomes harder and too brittle to reliably stretch at lower temperatures ( $< -10\text{ }^{\circ}\text{C}$ ) and soft and elastic at warmer temperatures ( $> 20\text{ }^{\circ}\text{C}$ ). This elasticity is beneficial for the decoupling of fragments of paint from the surface, however, when too warm the film would tear before it could be stretched sufficiently beneath the paint layer to release the fragment. Since the film needed to be handled considerably as part of the process, warming of the wax from body heat was inevitable and in many cases a great deal of the paint could not be removed from the film due to tearing and had to be disposed of. It may be possible to improve the efficiency of this approach with better temperature control of the wax film, however it is unclear exactly how this might be achieved, and perhaps more importantly, the need to further innovate on any approach using laboratory wax film is negated by how successful and comparatively effortless silicone rubber is as an application medium in contrast.

The most important step in efficiently producing microplastic paint particles is getting from layers of wet paint applied to a surface medium to solid unattached (pure) paint fragments. Once fragments of pure paint have been obtained, producing the particles is relatively simple since dried paint flakes are very brittle and can be easily broken by mechanical force to a size range which can be sieved. It is at this important step where the silicone rubber sheet offered the greatest benefit to efficiency over laboratory wax film. Unlike with the wax where careful stretching across a fingertip might decouple a  $\sim 1\text{ cm}^2$  paint flake from the wax, the entire silicone rubber sheet (surface area =  $1131\text{ cm}^2$ ) could be peeled away from the paint film leaving a single sheet of pure paint  $> 1000\text{ cm}^2$ , which was the case for P2. For P1, while this was not completely possible since this was more prone to tearing and curling, it was still possible

to decouple individual fragments of paint  $>100 \text{ cm}^2$ ; far larger than anything achievable using laboratory wax film and with considerably less time and effort investment from the user. However, this fact is primarily why P1 produced  $0.094 \text{ g cm}^2$  (36.5%) less than P2 using the same silicone rubber application material.

Despite the reduction in material efficiency for P1 compared to P2 on silicone rubber, production of P1 was more time-efficient ( $2 \text{ g h}^{-1}$ ; 14.8% increase). Interestingly, P2 production was (66%) more time-efficient than P1 on laboratory wax (although it should be considered that this 66% increase was just  $0.2 \text{ g h}^{-1}$ , given the overall poor performance of the wax method overall). Based on observations during the experiment, this is postulated to be due to the fact that P1 was slightly more brittle than P2 at the end of the drying period. This meant that fragments cracked more easily which made removal from laboratory wax more challenging, while on silicone rubber, since large flakes of paint were easy to remove for both paints, that extra brittleness made fragmenting and passing paint flakes through the 1 mm sieve easier. It is possible that a longer drying period than the allotted 14-day period would have benefitted P2, further increasing time-efficiency. Given this method is intended to stand as a proxy for any given paint particle production, it is likely worth adapting the drying period to best suit the paint being used in order to reduce user time and frustration during sieve separation. Drying time-efficiency is out of the scope of this study, however since paint drying is generally achieved through evaporation of the solvent, any approach to increase evaporation speed, such as temperature or airflow/ventilation increase [10], is likely to improve drying times, increasing time-efficiency of the process overall.

There are some limitations with the study. Just 2 different combinations of paint are tested on 2 different application materials without replicates. Because of this, it is highly likely that were the study to be repeated with different paints under differing conditions the efficiency rates are likely to vary. However, despite this limitation (and unlike the vast majority of scientific work), further research is not recommended. The difference in performance between the 2 application materials is so stark that, even with more variation afforded by different paints/combinations, it is extremely unlikely that laboratory wax would ever be even as close to optimal as silicone rubber for paint particle production. This does not mean that the entire method would not benefit for further refinement; active drying and cryomilling, for example, may well improve the overall efficiency from paint application to produced fragments, but simply in terms of application material, the efficiency of silicone rubber is so high that investing further research time into testing

other surfaces (and especially laboratory wax) is likely a waste of time and funds.

## Conclusion

Silicone rubber was  $\sim 36 \times$  more time-efficient than laboratory wax when used as application material for the production of paint particles. As such, laboratory wax should not be used as an application material, and silicone rubber is preferred. Indeed, despite not testing other application materials, the possibility is extremely limited for any given application material to perform better than silicone rubber, since the paint can simply be peeled away from the silicone rubber surface in seconds once cured. It is also inexpensive and widely available as it is used commonly in household baking. As such, for the production of paint particles for experimental purposes, silicone rubber is highly recommended as an application material.

## Acknowledgements

Not applicable.

## Author Contribution

AT did all the work, writing and funding acquisition.

## Funding

Open Access funding enabled and organized by Projekt DEAL. Funding was provided for this work as part of the PaintSed project was provided by the DFG (Deutsche Forschungsgemeinschaft) grant number TA 1720/1–1.

## Data Availability

All data generated or analysed during this study are included in this published article. The sources of all key materials used are also provided in the published article.

## Declarations

### Ethics approval and consent to participate

Not applicable.

### Consent for publication

Not applicable.

### Competing interests

The author declares that they have no competing interest.

### List of Abbreviations

Not applicable.

Received: 15 March 2023 / Accepted: 15 May 2023

Published online: 01 June 2023

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