

June 2024

## EPDLA's position paper on polymer dispersions, redispersible polymer powders made thereof and synthetic polymer microparticles

The European Polymer Dispersion and Latex Association (EPDLA), a Cefic Sector Group, is dedicated to promoting the safe manufacture, transportation, distribution, handling and use of waterborne polymer dispersions and redispersible polymer powders made thereof in compliance with regulatory requirements and industry guidelines.

EPDLA members are committed to Responsible Care<sup>®</sup> principles and have implemented risk management according to the precautionary principles.

On September 25, 2023 the European Commission published regulation (EC) No 2023/2055<sup>1</sup> amending Annex XVII to Regulation (EC) No 1907/2006 of the European Parliament and of the Council concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as regards synthetic polymer microparticles (SPM). The present position paper will discuss how this regulation relates to polymer dispersions and redispersible polymer powders made thereof.

#### **Polymer dispersions**

Polymer dispersions are used as raw materials (binders) in many waterborne applications, for example in adhesives, varnishes and coatings, printing inks, non-wovens, paper and paperboard textile finishing agents and redispersible polymer powders. Polymer dispersion technology has been used safely and successfully for more than 70 years and has contributed significantly to a reduction in the release of organic solvents into the environment. The use of polymer dispersions has helped to reduce the use of organic solvents in workplaces leading to improved worker health and similarly has contributed to cleaner air in the home, to an extension of the service life of many materials e.g. via surface protection, and efficient, durable and sustainable construction e.g. via contribution to insulation of buildings. Common to all dispersions and covered by this position paper, is a film forming process during application.

Polymer dispersions are mixtures as defined under Article 3(2) of the REACH Regulation<sup>2</sup>, consisting mainly of water and droplets containing high molecular weight polymers. Based on polymer weight and chemical nature, the polymer droplets are typically highly viscous. The size of such polymer

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<sup>&</sup>lt;sup>1</sup> Regulation - 2023/2055 - EN - EUR-Lex (europa.eu)

<sup>&</sup>lt;sup>2</sup> Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH), establishing a European Chemicals Agency, amending Directive 1999/45/EC and repealing Council Regulation (EEC) No 793/93 and Commission Regulation (EC) No 1488/94 as well as Council Directive 76/769/EEC and Commission Directives 91/155/EEC, 93/67/EEC, 93/105/EC and 2000/21/EC



containing droplets can widely vary between ca. <100 nm (<0.1  $\mu$ m) and 10,000 nm (10  $\mu$ m) in diameter<sup>3</sup>. For more details on the status of polymer dispersions with respect to nanomaterials, please refer to the EPDLA position paper on nanomaterials<sup>4</sup>.

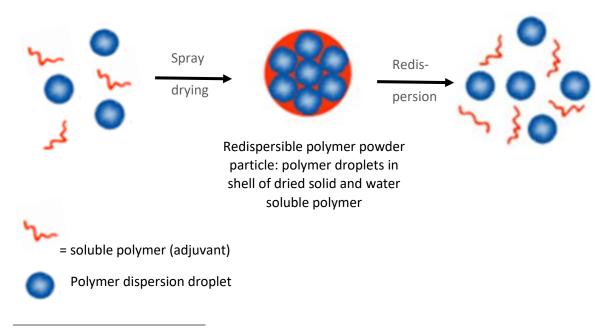
Within a dispersion, the polymer droplets are dispersed and stabilised in water and regarded as embedded in the liquid matrix. They cannot be isolated as discrete droplets or particles by simple separation techniques and do not exist without their waterborne environment. Their fate is to form a film e.g. as an adhesive or paint.

As the water in the mixture evaporates, a separation between the aqueous and the polymeric phase begins and leads to the film formation via coalescence of the polymer droplets<sup>3,5</sup>.

Human or environmental exposure to individual polymer droplets is thus not expected under advised conditions of use.

#### **Redispersible polymer powders**

Redispersible polymer powders (RDP) are obtained through spray-drying of polymer dispersions with the help of a spray drying adjuvant polymer. Such spray drying adjuvant polymers are solid and water soluble polymers. During spray drying larger particles in the micrometer range, typically >>1  $\mu$ m are formed as loose agglomerates of a few polymer dispersion droplets individually covered by the spray drying adjuvant polymer.



<sup>3</sup> nm = nanometer /  $\mu$ m = micrometer

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<sup>&</sup>lt;sup>4</sup> EPDLA position paper on polymer dispersions and nanomaterials (updated May 2023)

<sup>&</sup>lt;sup>5</sup> (469) EPDLA Sector Group of Cefic - YouTube



The RDPs are usually free-flowing fine powders, with a particle-size distribution in the micrometerrange. Moreover, the RDPs contain a sort of liquid core (the original dispersion polymer droplets) in a shell of a dried solid (containing also inorganic material) and water soluble polymer. In contact with water, the redispersible polymer powder particles disappear as the soluble polymer dissolves and release the individual dispersion droplets in their original state again. This redispersed polymer droplet has the same properties as the original polymer dispersion droplet.

Due to their decisive influence on cement, lime or gypsum based finished drymix mortar products, as well as on cement free and mineral-binder free systems, redispersible polymer powders are mainly used as building material additives in construction-applications to provide e.g. adhesion to many types of surfaces and substrates, flexibility or waterproofing.

#### Polymer Dispersions and synthetic polymer microparticles

According to the regulation SPMs are polymers that are solid and which either:

(a) are contained in particles and constitute at least 1 % by weight of those particles, or (b) build a continuous surface coating on particles.

EPDLA has concerns around the methods proposed for assessing whether in water dispersed polymeric matter falls under the synthetic polymer microparticle definition.

EPDLA suggests to use the Minimum Film Formation Temperature (MFFT)/White point instead, which is a common industrial ISO standard method.

The three test methods mentioned in Annex No. 2. (d) of regulation (EC) No 2023/2055 for assessing the state of matter are not applicable to polymer dispersions (most of the polymers in such dispersions are amorphous matter and therefore do not have a melting point. ASTM D 4359-00 or the penetrometer test can only be applied to bulk polymers). Since polymer dispersions are always placed on the market in a homogeneous liquid water phase, their state of matter cannot be assessed using these methods. Therefore, other criteria are needed to determine if in water dispersed polymeric matter falls under the synthetic polymer microparticle definition.

The polymer droplets of a typical polymer dispersion are dispersed and stabilized in water by hydrophilic polymers and/or surfactants and regarded as embedded in the water matrix. The evaporation of water creates high pressure by capillary forces between the droplets which leads to the fusion of the polymers when the Minimum Film Formation Temperature (MFFT) is below room temperature<sup>5</sup>. Any crosslinked or very high molecular weight polymer molecules are diffusely (no interface) distributed within that droplet. The outer shell of droplets consists typically of polymer molecular weight polymers interact strongly with the typically present emulsifier to stabilize the droplet in water. This excludes typical polymer dispersions to fall under the SPM definition: The droplets are not considered solid particles, nor do they form a continuous surface coating on particles.

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The above clarifications apply both to individual substances and mixtures where dispersion polymer droplets may be present at a concentration  $\ge 0.01\%$  w/w, such as, for example, in adhesives or paints.

While most polymer dispersions cannot be considered SPM, there may be polymer dispersions that could be considered SPM due to their high minimum film formation temperature.<sup>6</sup> For these cases, however, derogations will apply, as described in the following: Applications of polymer dispersions involve the formation of a film. Film formation is critically dependent on the minimum film forming temperature (MFFT) /white point of the polymer dispersion, which is the lowest temperature at which a thin layer of a polymer dispersion will still dry to form a coherent film. The white point temperature would thus be comparable to an initial melting point as defined in Annex XVII of the regulation under point 2d(i). Since continuous, uniform film formation, as prescribed by the MFFT, is not a must for deciding whether the polymer dispersion falls under the SPM definition, the so-called white point could also be used here: This is the temperature at which coalescence of the polymer droplets into a polymer film begins and at which an opaque film changes into a transparent film, which is a clear indication of the presence of liquid material.

Both MFFT and white point can be measured according to the ISO 2115 standard. The minimum film forming temperature (MFFT) is a widely used and extremely important application relevant property of polymer dispersions (see above). The white point temperature describes much better the coalescence temperature of polymer dispersions droplets and should therefore be used for the microparticle classification in certain cases (i.e. near room temperature). In many cases, when the MFFT is clearly below room temperature, the use of the MFFT will suffice.

We ran an anonymized Round Robin (RR) or Ring Test between EPDLA members to validate the MFFT method as industrial standard. The MFFT method has been used by the industry for many years. The standard deviation of <1.5°C that was finally achieved in the RR substantiates the accuracy and practicability of the MFFT method as industrial standard. Details of this round robin test can be found in Annex I.

#### Labelling and reporting requirements are even foreseen for applications that are out of scope

Paragraphs 4 and 5 of the draft synthetic polymer microparticle restriction provide derogations for the placing on the market of SPM or mixtures containing such microparticles if:

- they are used at industrial sites;
- or if the resulting film does not meet the definition of a synthetic polymer microparticle;
- or if the microparticles are permanently incorporated into a solid matrix at the time of use,

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<sup>&</sup>lt;sup>6</sup> Even with high-MFFT polymers, coalescence can be forced by adding so called coalescence agents. This is typically done by downstream users, e.g. paint manufacturers.

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• or if the microparticle is contained by technical means so that no release to environment can occur.

We therefore understand that the use of polymer dispersions for most applications will not be impacted by the restriction for placing on the market under this regulatory proposal. However, the European Commission foresees labelling and reporting requirements which comprise among others: Suppliers of aforementioned derogated products containing SPMs shall provide instructions for use and disposal to avoid releases of SPMs to the environment as well as information on quantity or, as applicable, concentration of SPMs in the substance or mixture.

#### Redispersible polymer powders and synthetic polymer microparticles

The RDPs are usually free-flowing fine powders, with a particle-size distribution in the micrometerrange. The RDP particles contain the original Polymer containing droplets in a shell of a dried soluble polymer. They exist as a mixture of two polymers, the spray drying adjuvant polymer and the dispersion polymer.

Since the spray drying adjuvant polymer has a solubility of much greater than 2g/l, it is excluded from the restriction. The second polymer coming from a dispersion is not considered as SPM if the MFFT is lower than room temperature (ca. 20°C). In most cases redispersible polymer powders therefore do not fall under the SPM definition. When the redispersible polymer powder is dispersed in water the individual dispersion droplets are released in their original state again and don't fall under the SPM definition if the MFFT or White Point Temperature is below 20°C.

# Are redispersible polymer powders concerned by 'Operation Clean Sweep' or the proposed Regulation of the European Parliament and of the Council on preventing plastic pellet losses to reduce microplastic pollution?

Operation Clean Sweep (OCS<sup>7</sup>) is a voluntary industry initiative to minimize plastic pellet loss. The term pellet covers either resin pellets, flake or polymer *powders*. However, the powders covered by OCS are meant for the manufacturing of plastic, whereas redispersible polymer powders go into totally different applications like for example in concrete or mortar formulations. Furthermore, the outer shell of such RDPs is soluble and the "inner core" contains polymer droplets.

This pellet definition of Operation Clean Sweep contrasts the Proposal for a Regulation of the European Parliament and of the Council on preventing plastic pellet losses to reduce microplastic pollution COM(2023) 645<sup>8</sup>, where "plastic pellet" means "a small mass of preformed polymer-

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<sup>&</sup>lt;sup>7</sup> Operation Clean Sweep<sup>®</sup> Progress Report 2019 - Plastics Europe DE

<sup>&</sup>lt;sup>8</sup> <u>Microplastics pollution – measures to reduce its impact on the environment (europa.eu)</u>

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containing molding material, having relatively uniform dimensions in a given lot, that is used as feedstock in plastic product manufacturing operations".

Latter definition clearly does not comprise redispersible polymer powders, as they are not intended for molding.

Therefore, EPDLA sees RDPs not concerned neither by the OCS initiative nor by proposed regulation COM(2023) 645.

#### **National regulations**

EPDLA is aware of national regulations, for example in France, to regulate Microplastics. In the case of the French restriction on the use of Microplastics (art. 82, AGEC law), which entered into force January 1st, 2023, it is impossible at the current stage to comment on the regulatory proposal due to the lack of definitions. French Chemical industry Associations actively advocate for a harmonization of the French regulation on Microplastic and the European regulation on SPM.

#### Conclusion

The EPDLA members conclude that any polymer dispersion with a white point or MFFT below room temperature (around 20°C) does not meet the criteria of synthetic polymer microparticles (SPMs) due to the liquid nature of the polymer chains (droplets), which enables formation of a continuous film when water evaporates.

Redispersible polymer powders (RDP) consist typically of two different polymers: a solid but water soluble polymer which forms the "outer shell", and a few inner dispersion polymer droplets. The solubility of the soluble polymer part is usually much greater than 2g/l. The "inner core" typically consists of a dispersion polymer with MFFT < room temperature, which means, this polymer has not to be considered as SPM. Therefore, both types of polymers in a RDP and thus the redispersible polymer powder as such do not meet the criteria for SPMs.

#### Disclaimer

- The present position paper has been developed by EPDLA members in good faith, to the best of its knowledge and following the latest scientific evidence.
- The position paper is offered to all EPDLA members for further use. Each producer might add additional information in the communication towards customers, depending on the specific situation.
- EPDLA commits to update this document in view of any new relevant available information.







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#### For more information please contact:

Eleonora Mancinelli, Sector Group Manager, EPDLA, + 32. 492345072 or <u>elm@cefic.be</u>.

#### About EPDLA

EPDLA (European Polymer Dispersion and Latex Association), a Cefic Sector Group founded in 1991, is dedicated to promote the safe manufacture, transportation, distribution, handling and use of waterborne polymer dispersions, in compliance with regulatory requirements and industry guidelines. EPDLA members are committed to Responsible Care<sup>®</sup> principles and have implemented risk management according to the precautionary principles.

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#### **ANNEX I: Round robin on MFFT measurement**

The MFFT was measured by 10 EPDLA member companies on four different commercially available latex samples in a series of Ring- or Round Robin tests (RR). The procedure applied was based on the DIN ISO Norm 2115. The image below shows the films of the samples B to E coated on a film bank with a temperature gradient from 10°C to 30°C. The transition from a homogeneous film without cracks indicates the MFFT and were the film start to be white indicates the White Point Temperature. Since the length of such film bank is 50 cm, the temperature gradient of 20°C over this length corresponds to 2,5cm per 1°C, which allows precise and repeatable results.



Figure 1 Film Bank coated with samples B to E

Before starting the RR the member companies agreed on some settings and procedures which are covered by DIN ISO 2115 and might have a significant impact on the accuracy of the results. The table below shows the analyzed parameter and some recommendations in the raw Harmonized procedure. However, the method turned out to be very robust and the most important considerations are the equilibration time of the film bank until a stable temperature gradient is achieved and a sufficient drying time of the latex film of three hours at least.

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Table 1 Recommended settings and procedures for the MFFT and White Point Temperature measurement

	Explanation	Harmonized Procedure
Instrument	Manufacturer, Model	
Method/ Standard	according DIN ISO Nom	
Instrument dimensions	length width of the tempearture gradient bank	50cm X 20cm
Film application	Blade, thickness, widthor gatter, depth, width?	100 µm
Temperature Range	i.e. from 5°C to 25°C	10°C to 30°C
Randomization of repeats	Measured films in a row or a special design	All repeats should be measured on different runs, at least three repeats
Determination MFFT	visual, with a pen, or??	report repeat with one digit of 0.5 at least, average will be reported as integer, visually when cracks start, first crack
Determination white point	visual, with a pen, or	report repeat with one digit of 0.5 at least, average will be reported as integer, visual, the borderline from white to transparent
Equilibrium time of the bench	Time before apply the film	after 1h or equilibrium is reached
Film forming time on the bench	Time after film application and drying before determing the MMFT	Minimum of 3h drying time
Substrate	Alumina foil, plastic foil,	
atmosphere	Air, nitrogene, air flow, open or closed cabine	

The diagram below shows the results of all 10 participating member laboratories. All samples were measured up to six times and 3 times at least. ISO 2115 requests a Standard Deviation (SD) below 2°C for the results. The RR shows a SD below 1,5°C for the repetition of measurements of the same latex (Figure 3) and between the companies (Figure 4).

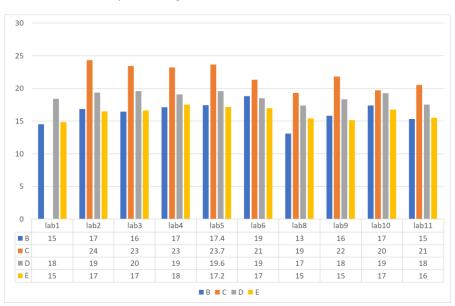


Figure 2 MFFT results

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Figure 3 Standard Deviation of the MFFT results for all labs and samples

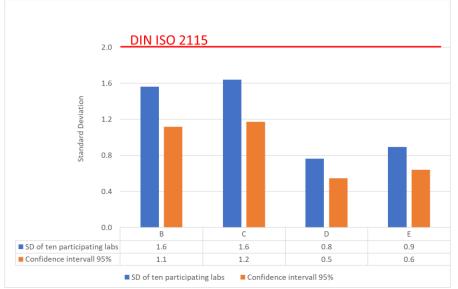


Figure 4 Standard Deviation and confidence level of the MFFT results between the company labs

The Z-Score approach was applied to quantify the difference of each company to the average of all measurements. The Z-Score expresses the deviation of results in magnitudes of the SD for a lognormal distribution. The deviation of the results from the average should be between 2 and 3 at least to fit to a lognormal distribution.

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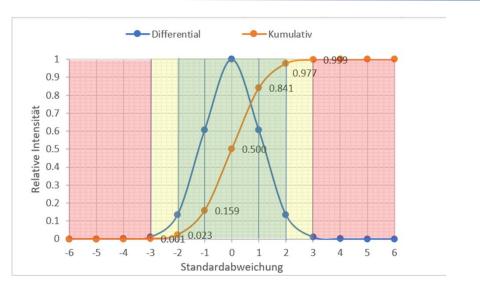


Figure 5 Lognormal Distribution with indication of the 68,2, 95,4% and 99.8% confidence interval

The Diagram below shows the Z-Score of all results for a onefold SD of 1.0°C. Most of the results deviate from the average below 2 SD (+/-2°C) and at least below 3 SD (+/-3°C).

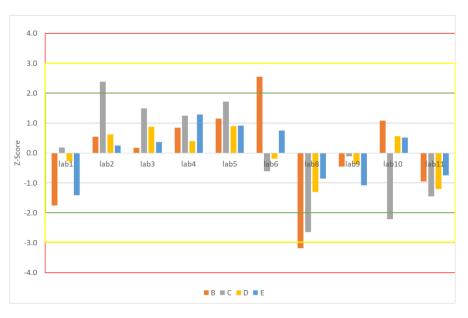


Figure 6 Z-Score of the results for a onefold SD of 1.0°C

The Diagram below shows the Z-Score for the average MFFT of the four samples measured by each company lab. The deviation between the company labs lies between 2D or +/-2°C.



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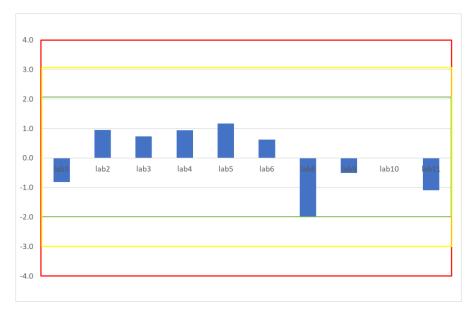


Figure 7 Z-Score for the average of four samples

#### Conclusion

The results of this ring test show that the inter-laboratory consistency of the MFFT measurement results is very good (<1.5°C standard deviation), when the recommendations of DIN ISO 2115 are followed. This high level of fit supports the reliability of this MFFT method for measurements of polymer dispersions in the context of SPM regulation.

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