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facts

Boosting sustainability in architectural paints – CITROFOL® BI as bio-based coalescing agent

Introduction

Coalescing agents in coating formulations

In the coating industry, there is an increasing demand for safe, environmentally friendly products. This trend is mainly being driven by increasingly stringent regulations, especially in Europe. These regulations require volatile organic compounds (VOCs) and CO_2 emissions to be reduced as well as the replacement of critical raw materials by compliant alternatives. At the same time, the coatings performance and aesthetics need to be maintained. Bio-based raw materials have been found to be appropriate substitutes, frequently carrying significantly smaller carbon footprints than their fossil counterparts. Many of them also have better toxicological profiles and are biodegradable. Coatings producers have thus already begun taking steps to replace various ingredients in their formulations with more sustainable alternatives.

In water-based coating formulations, the coalescing agent is an important component which significantly influences film formation during the drying process and therefore the quality of the coating. Conventional coalescing agents evaporate from the coating during and after drying. This process depends on the volatility of the coalescing agent, which is related to its boiling point. A low boiling point enables the coalescing agent to evaporate more quickly but may also lead to it being considered a VOC.[1] Furthermore, all volatile coalescing agents contribute to emissions in the surrounding environment and therefore require specific safety precautions.[2,3]

On the other hand, CITROFOL® citrate esters are non-VOC and comply with the latest toxicological and environmental safety requirements. Being a permanent coalescing agent, they remain in the coating. They have excellent toxicological and eco-toxicological profiles, are very versatile, and compatible with numerous polymers. Additionally, CITROFOL® citrate esters are up to 100% bio-based, depending on their specific type.

In a previous study[4], we demonstrated the usability of CITROFOL® BII as a coalescing agent in architectural paint formulations. In the present article, this study was broadened to CITROFOL® BI as it additionally features a higher bio-based content.

The performance of CITROFOL® BI was compared to two alternative coalescing agents available on the market. Coalescing agent 1 is an ester alcohol and classified as a temporary coalescing agent that will evaporate over time. Coalescing agent 2 is an ester mixture that, like CITROFOL® BI, is expected to remain in the dried film.
An overview of the physiochemical characteristics of all tested candidates can be found in table 1.

Table 1: Overview of relevant physiochemical characteristics of tested coalescing agents

Properties	CITROFOL® BI	Coalescing agent 1	Coalescing agent 2
Boiling Point	309°C	254°C	290–310°C
VOC	No	Depends on regulation	No
Chemistry	Ester	Ester alcohol	Ester
Water Solubility	Insoluble	0.1% soluble	Insoluble
Sustainability	100% bio-based	Not bio-based	Not bio-based

This selection of coalescing agents enabled us to present further valuable insights into the performance of CITROFOL® BI as coalescing agent.

Materials and methods

The test matrix for this study was an architectural paint formulation. Three batches were produced in the lab, each containing one of the aforementioned coalescing agents. The coalescing agents are needed because the dispersion has a Minimum Film Forming Temperature (MFFT) of 20°C. The formulation is shown in table 2.

Table 2: Architectural paint formulation

Trade Name	Description	[wt%]
Water deionized	Solvent	21.15
Natrosol™ 250 HR	Hydroxyethyl cellulose	0.40
Monoethanolamine (MEOA)	pH adjustment	0.05
Dispex® AA 4140	Dispersing agent	0.60
Tego® Foamex 1488	Defoamer	0.15
Carbowet® GA-100	Grinding aid	0.15
Tioxide® TR81	Titanium dioxide	19.0
Omyacarb® 2 GU	Calcium carbonate	9.00
Omyacarb® 5 GU	Calcium carbonate	14.0
Acronal® S 790	Styrene acrylic dispersion	31.5
Tego Foamex® 1488	Defoamer	0.15
Acrysol™ RM-825	Synthetic thickener (HEUR)	1.45
Coalescing agent	See Table 1	2.40
	TOTAL	100

Application characteristics

Rheology

To assess flow behaviour, a flow curve of each paint batch was measured (twofold measurement). The flow curve was measured by rotational viscometry (Haake RheoStress, parallel plate geometry P20 CS L). Because of the samples shear thinning behaviour, they were allowed to rest for a period of one minute after application onto the rheometer platform before measurement was started. Apparent viscosity was determined at continuously increasing shear rates from 0.01 to 100 s^{-1}.

Leveling

A special applicator was used, enabling two parallel lines of the coating to be applied at increasing film thicknesses from 250 µm to 4000 µm. The test chart was kept horizontal while the coatings dried. After drying, observations were made of the film thickness above which the distance between the two lines started to narrow. This was defined as the measure of leveling.

Sagging

A special applicator was used, enabling parallel lines of the coating to be applied at increasing film thicknesses from 75 µm to 300 µm. Immediately after application, the test chart was placed in a vertical position and left to dry. After drying, observations were made of the film thickness above which the coating showed signs of sagging, such as dripping. This was defined as the measure of sagging.

Drying time

Drying was monitored by tracking weight loss over time. This corresponds to the evaporation of any liquid component of the coating formulation. The drawdowns were stored at room temperature. An analytical balance was used to ensure sufficient precision.

Surface properties

Pendulum damping

To evaluate the hardness of the coatings, König pendulum hardness tests were conducted in accordance with ISO 1522:2023 using a BYK Instruments byko-swing. The number of swings needed to damp the pendulum from 6° displacement down to 3° displacement was counted to assess this property.

Gloss

Gloss values were determined in accordance with ISO 2813:2015 using a Gloss Haze DOI Meter from Rhopoint Instruments GmbH. Classification was undertaken according to the limits given in DIN 13300:2023.

Blocking resistance

In accordance with EN 297, a 150 µm thick layer of the coatings was applied to a Leneta card and left to dry for 24 hours at ambient temperature. The coatings were then stacked with the coated sides facing each other and were placed under 1 kg of weight for 24 hours. Once the test was complete, the coatings were classified according to the force needed to separate the layers, ranging from a1 (no adhesion at all) to a5 (inseparable).

Wet-scrub resistance

In accordance with ISO 11998:2006, the coatings were applied to special test charts and left to dry for 28 days in an air-conditioned environment. A defined washing liquid was applied to the surface of the coatings, and then 200 strokes were performed using an abrasive pad from Jost (type S1200/E) in a TQC Sheen Scrub from Industrial Physics. Classification was undertaken according to the limits given in DIN 13300:2023.

Yellowness Index

In accordance with ASTM E313-73, the Yellowness Index was measured using a CM-600d spectrophotometer from Konica Minolta. The value on the index increases with the yellowness of the coating.

Storage stability

Since storage stability is a crucial property for paint manufacturers, the pH value of all samples at room temperature was measured over a period of 84 days using a standard pH meter.

Besides being stored at room temperature, all coatings were also subjected to accelerated storage at 40°C for 3 months. After this period, they were inspected to identify any phase separation, which may indicate insufficient storage stability.

Results & Discussion

Application characteristics

All paint batches had a high initial viscosity, ranging from approximately 850 Pa*s up to almost 900 Pa*s. Increasing shear force led to a decline in viscosity, as is to be expected for architectural paints. This property enables the coating to be both storage stable and easy to apply. There was good conformity among the batches, as shown in figure 1.

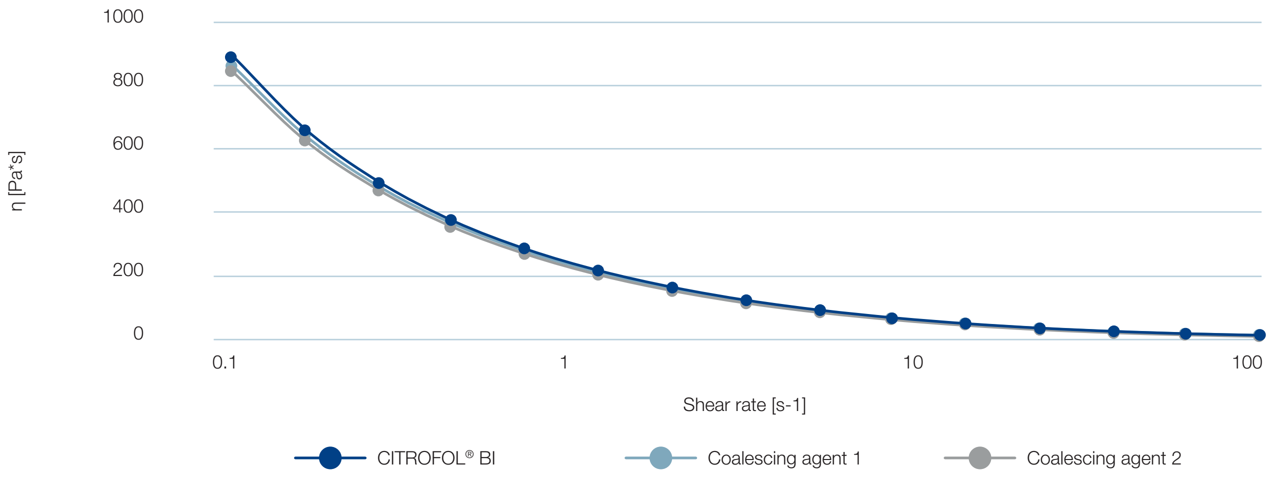

Figure 1: Flow curves of all paints after 24 hours storage

None of the tested samples showed any signs of leveling or sagging. This is in line with the high viscosity in the absence of shear force. If desired, the amount of synthetic thickener in the formulation could be reduced to achieve a smoother optical experience.

Contrary to widespread concern, the use of a permanent coalescing agent did not lead to longer drying times. As shown in figure 2, all three batches were fully dry and thus ready for another coat to be added after 6 hours.

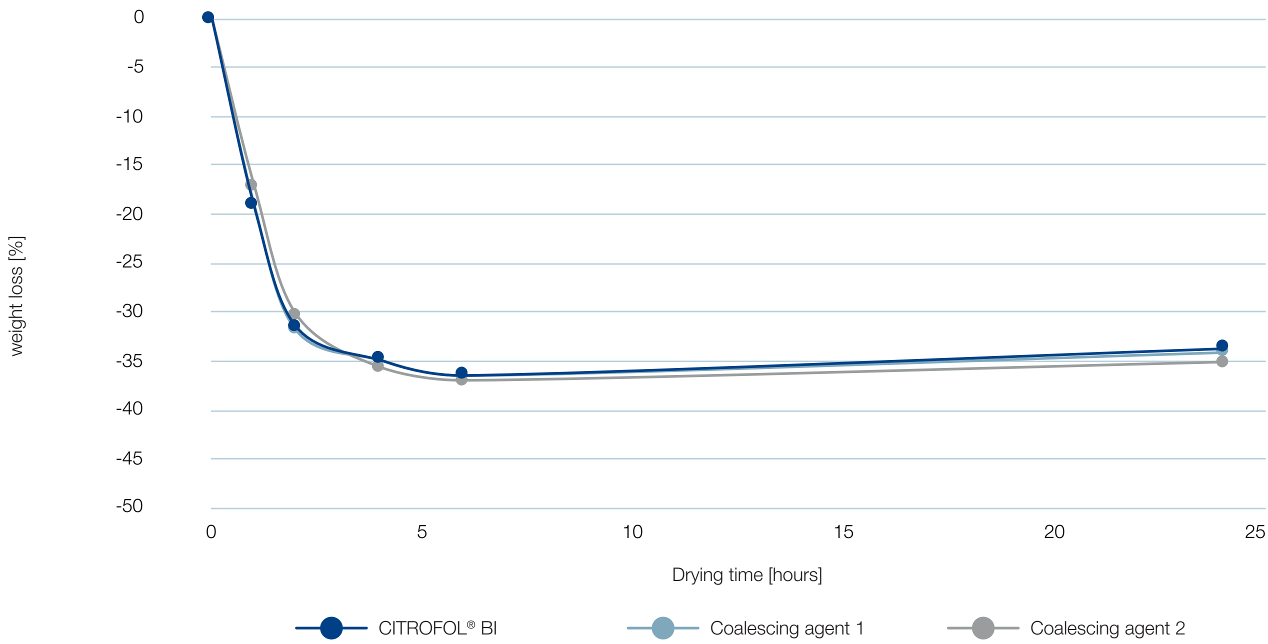

Figure 2: Weight loss of applied coatings containing different coalescing agents

Surface properties

Pendulum damping

To detect the coalescing agents influence on surface hardness, the pendulum damping was measured repeatedly over the course of 28 days. While the initial values after 24 hours were similar, significant differences became apparent over time. Coalescing agent 1, being a VOC with a lower boiling point, steadily evaporated. This led to an increase in surface hardness. CITROFOL® BI in comparison maintained a steady level of pendulum damping. These values were more consistent than those of Coalescing agent 2 (another ester), which exhibited some evaporation at the end of the measurement period. However, all the paint formulations were very soft coatings, as is typical for architectural paints. The slightly lower values produced by permanent coalescing agents are therefore not a concern.

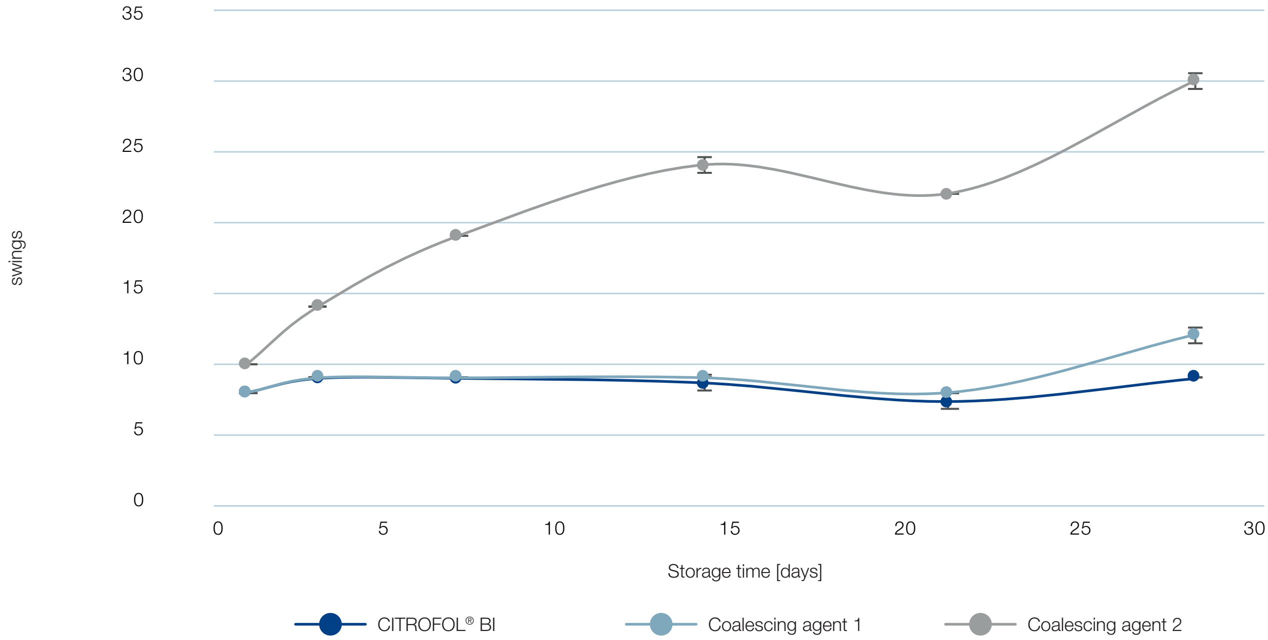

Figure 3: König pendulum damping of coatings containing different coalescing agents

All paints achieved the same results for gloss, block resistance and wet-scrub resistance. They had gloss values less than or equal to five gloss units, which is considered dead matt. For blocking resistance, an a1 rating was achieved. This indicates slight adherence, while needing no effort to separate the specimens. In terms of wet-scrub resistance, all samples conformed to class 1, indicating a mean loss in coating thickness no greater than 5 µm.

Although there was no visual difference to the naked eye, the yellowness measurements proved that there was close to zero yellowing with CITROFOL® BI, a smaller change than with either of the other coalescing agents tested. However, low values were obtained for all formulations, as can be seen in figure 4.

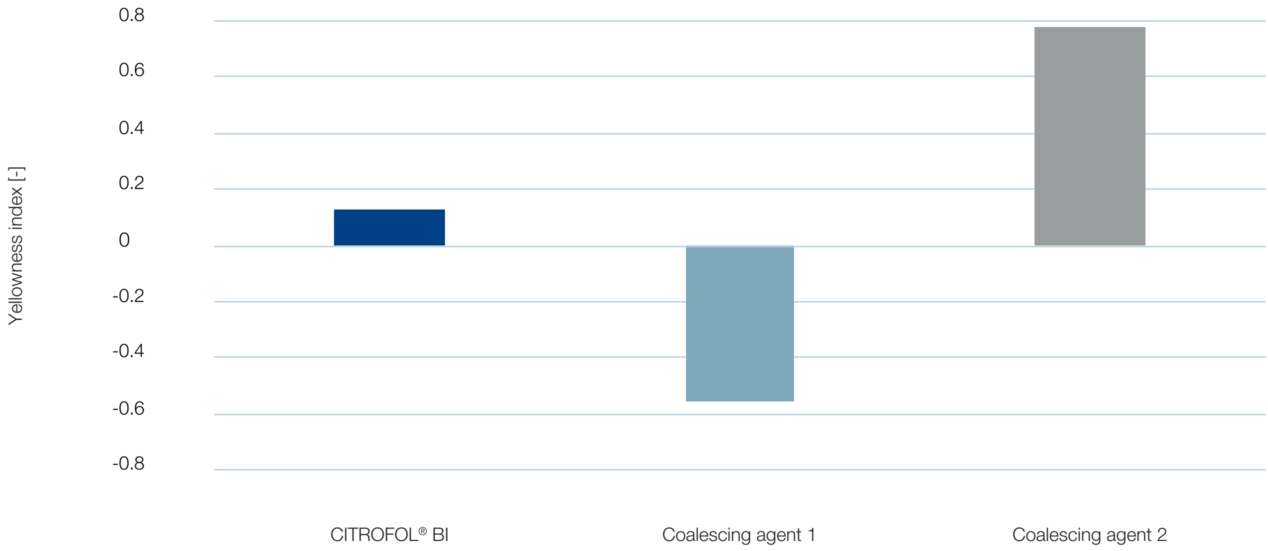

Figure 4: Yellowness Index of coatings containing different coalescing agents

Storage stability

pH value over time

The pH value of approximately 8.7 remained mostly stable within all paint batches over the 3-month storage period. This consistency indicates that the formulations did not destabilise in-can.

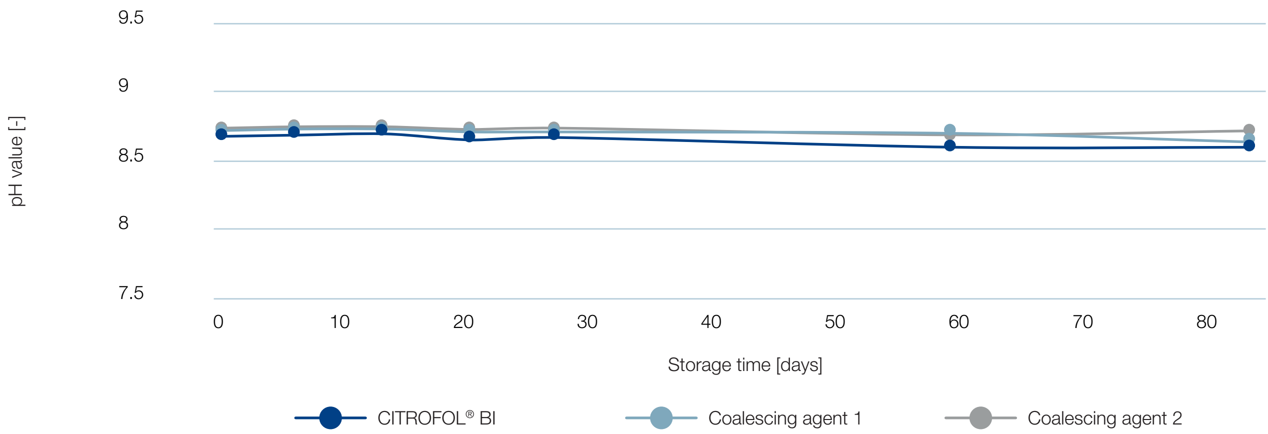

Figure 5: pH values of coatings containing different coalescing agents when stored at room temperature

Evaluation of syneresis

Even after three months storage at 40°C, none of the samples showed any signs of syneresis.
This can be considered proof that the formulations are sufficiently stable.

Conclusion

Our extensive tests confirm that CITROFOL® BI is an excellent coalescing agent in architectural coatings. CITROFOL® BI efficiently combines good film formation with a non-toxic nature and a 100% bio-based ingredient, rendering the paint safer and more environmentally friendly. The effects of CITROFOL® BI on coating properties are virtually identical to those of conventional coalescing agents, so it can be used as a drop-in replacement to formulate more eco-friendly paints with minimal effort.

References

[1] Directive 2004/42/CE of the European Parliament and of the Council of 21 April 2004 on the limitation of emissions of volatile organic compounds due to the use of organic solvents in certain paints and varnishes and vehicle refinishing products (accessed 16.07.2021).

[2] C. E. Stockwell, M. M. Coggon, G. I. Gkatzelis, J. Ortega, B. C. McDonald, J. Peischl, K. Aikin, J. B. Gilman, M. Trainer, C. Warneke, Volatile organic compound emissions from solvent- and water-borne coatings – compositional differences and tracer compound identifications. Atmospheric Chemistry and Physics, 2021, 21:6005–6022.

[3] https://www.epa.gov/indoor-air-quality-iaq/volatile-organic-compounds-impact-indoor-air-quality (accessed 20.01.2025).

[4] K. von Nessen, K. Klingberg, facts – CITROFOL® as coalescing agent, 2019.

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