

Getting the most out of Oxylink™

Dr. Marc Herold, Dr. Steffen Pilotek, Bühler Group.

The evaluation of Oxylink will always be carried out according to the project in question. Because adding Oxylink into an already optimized formulation may not reveal the full potential of this additive, the authors suggest alternatively to start the evaluation of Oxylink at the polymer level and to build subsequently an improved formula. This paper contains an overview of Oxylink and it suggests an initial test protocol for a step-by-step evaluation of the additive.

Oxylink is an additive used to improve performance properties of waterborne resin systems that are related to cross linking. The product can be used in formulations of paints, coatings, or sealants and it is not limited to a specific substrate, film transparency, gloss regime, application or curing process of an application.

Oxylink benefits.

Oxylink increases cross linking and related performance benefits. Cross linking can be characterized by MEK double rub resistance according to ASTM D 5402. Compared with the same formula without Oxylink, the additive typically increases MEK rub resistance by a factor of 2 – 8. Related performance benefits include alcohol, water and humidity resistance. In addition, blocking resistance and in turn tackiness and sandability can be improved by Oxylink. The additive can also increase tensile strength and glass transition temperature T_g without impact on elongation of elastomeric polymer compositions.

Moreover, Oxylink can result in an accelerated drying characteristic as it acts quickly within the drying film. The film stability in terms of MEK rub, water, blocking or other resistance properties is higher already after a shorter drying time resulting in better “early” resistance properties. Oxylink has also been shown to increase pendulum hardness in alkyd emulsions, especially in conjunction with cobalt driers.

Oxylink chemistry.

Oxylink is a formulated product based on ZnO nanoparticles dispersed in water. While the general benefit of ZnO in waterbased formulas has been recognized in the past, making use of this benefit has often proved challenging for several reasons, many of them related to efficiency and compatibility. Oxylink has been formulated with a specific focus on improved efficiency and compatibility. It uses nanoparticles as ZnO source which provides a large active surface area already at a low use level. The

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particles are surface modified to provide optimized reactivity and stability in water while additional components act synergistically and improve compatibility so increased performance can be obtained at retained formula stability. These characteristics allow Oxylink to be used to increase cross linking-related performance in 1-component formulations. Oxylink is mild in pH (8.5), almost odorless and virtually VOC free.

Compatible polymer chemistry.

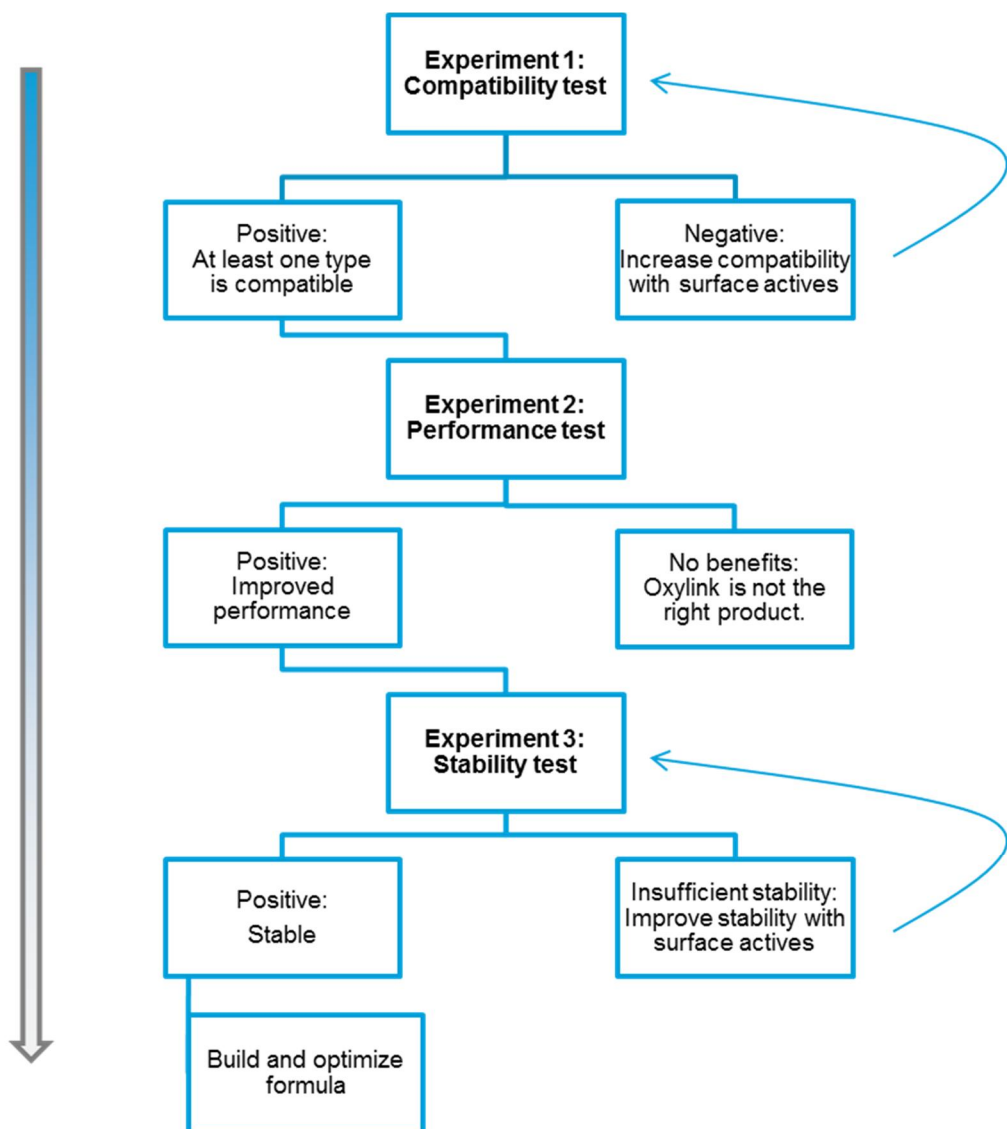
The particles in Oxylink are anionically stabilized and the product is compatible with anionically stabilized polymer emulsions. Oxylink is not compatible with cationic polymers, this combination will result in destabilization and flocculation. It has been proposed that the increased cross linking results from a chemical interaction of carboxylic groups with the ZnO surface provided by Oxylink. Consequently, compatible polymers need to provide carboxylic groups (neutralized acid functionality) for interaction with Oxylink. A large number of anionic acrylic resins including 100% acrylics, styrene acrylics, self-cross linking acrylics and acrylic-PUD hybrids have been successfully used with Oxylink. In addition, a smaller sample of PUD and alkyd emulsions have also been evaluated and shown benefits with Oxylink.

Available product types and differences.

Oxylink is available in three types (Oxylink 3101, 3102, 3103) that differ in composition to allow for a broader range of applications. As excellent compatibility with a formula is important for the performance of Oxylink, variations in the composition are provided that can result in a better match with a given formulation or polymer emulsion. While there are no quality differences between the Oxylink types, the compositions show different physical properties, e.g., apparent viscosity η ($\eta_{3101} < \eta_{3102} < \eta_{3103}$) and solid content c ($c_{3101} > c_{3102} > c_{3103}$). Due to the broad variety of formulations and their resulting chemical and physical characteristics, we recommend a set of simple lab tests to identify the most effective Oxylink type in a given formulation.

Test procedure.

Bühler recommends starting the evaluation of Oxylink by screening the three additive types in a simple test protocol of 3 experiments to identify the most promising type. Further optimization work can then focus on one instead of 3 products, which greatly minimizes the formulation effort. As the benefits from Oxylink are due to the interaction between the Oxylink actives and the polymer system, Bühler suggests to start the Oxylink evaluation on the polymer level. The following flow diagram provides a quick overview of the suggested experiments.



Experiment 1:

Four samples of 100 g polymer emulsion each are provided . Each Oxylink type is agitated well, e.g., by shaking the container. 1 g Oxylink 3101 is added to the first, 1 g of Oxylink 3102 to the second and 1 g of Oxylink 3103 to the third sample. The fourth sample is kept for comparison. Each sample is mixed well. The appearance of each sample is evaluated.

Ideally, the appearance of the samples remains unchanged. No flocculation, sedimentation, or notable change in viscosity should be observed. Particular attention should be given to any difference between the Oxylink types. For example, when the acrylic emulsion Neocryl A 622 is evaluated according to this procedure it turns out that Oxylink 3103 is not compatible with the binder. It is recommend to continue the evaluation with Oxylink types 3101 and 3102.

Typically, at least one of the Oxylink types is compatible with the polymer under evaluation. If necessary, the compatibility of Oxylink and a polymer emulsion may be improved by addition of a small amount (1 – 5% on Oxylink) of surface active components such as 2-amino-2-methyl-1-propanol (“AMP”) or other additives specified in the technical data sheet. As an example, a mixture of the self-cross linking acrylic emulsion Neocryl XK 98 with Oxylink tends to sedimentation. Adding 1 – 3 % (m/m) of AMP to Oxylink 3103 before adding 1% of this mixture to Neocryl XK 98 yields a compatible formula.

Experiment 2:

A film from each compatible sample from experiment 1 is drawn onto a glass slide. This experiment may require addition of some coalescent to achieve film formation. The appearance of each drawdown is evaluated with respect to haze and clarity. Each drawdown is subjected to a performance test. The test should be relevant for the project at hand and at the same time it should be quick and simple enough as suitable for a screening experiment. Suggestions include:

- a) MEK rub resistance test: A higher value until film removal indicates higher cross linking.*
- b) Water or alcohol spot test: Higher cross linking may lead to less damage and less swelling.*
- c) Tackiness: The tackiness of the film may be evaluated by paper adhesion or by touch.*
- d) Drying time: The drying characteristic may be monitored e.g., using a dry time recorder.*

While Oxylink contains ZnO particles, the particle size is small enough so Oxylink can be used in clear coatings even over dark substrates. Better clarity and lower levels of haze in a drawdown typically indicate better compatibility between Oxylink and polymer and therefore the film with the highest clarity and lowest haze is expected to perform best.

This prediction is verified in a simple performance test. As an example, a drawdown of acrylic emulsion Craymul 2166 resists 20 MEK double rubs without Oxylink and 58 double rubs when adding 1% Oxylink 3101 while other Oxylink types result in a film resisting ca. 50 MEK double rubs.

Experiment 3:

The liquid samples from experiment 2 are stored at room temperature or at elevated temperature overnight.

The samples should remain stable also under storage conditions. While Oxylink generally shows excellent compatibility characteristics, Bühler recommends testing the storage stability of the final formulation according to product/ project requirements. In this screening, the focus is on the differences between Oxylink types for which a shorter wait time is sufficient. As with compatibility in experiment 1, the storage characteristics of Oxylink may be improved if required by adding some AMP or other surface active components specified in the technical data sheet (i.e. Disperbyk 191, Surfynol 231, or Borchigen 650).

The result of this test protocol is a combined performance and stability ranking of Oxylink types for a particular resin system. The best candidate can now be used to build an improved formula.

Further Formulation work.

While Oxylink acts on the emulsion polymer, additional components of a formulated coating may influence the benefit.

Notable components generally comprise coalescents, surfactants and other surface active components as well as pigment preparations, and thickeners. Following a previous example, adding 1% DPnP to a resin sample of Craymul 2166 containing 1% Oxylink 3101 reduces the MEK double rub resistance of the film from 58 to ca. 32, while using 1% Texanol instead results in 49 double rubs and using 1% DPM provides a film that withstands 62 double rubs. Bühler therefore recommends to check the performance benefits after addition of further formula components. A discussion of the influence of such formulation components will be the topic of future work.

As a final step, Bühler recommends to carry out a ladder study to establish the optimum concentration of Oxylink in a formulation. In most cases, focusing on the range from 0.3 – 1.5% Oxylink will help identifying the optimum additive concentration.