



# TECHNICAL INFORMATION ADDITIVES FOR PIGMENT CONCENTRATES

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# **Pigment concentrates**

Pigment concentrates are widely used as intermediates to tint paints or plastics. When producing and utilizing pigment concentrates, multiple requirements must be fulfilled to achieve optimum performance:

- Producability/controllability during production
- Reproducibility/stability of quality
- Storage stability of the pigment concentrates
- Compatibility in different systems
- Optimum performance in final application
- No negative impact on other parameters and properties

This brochure provides to give an overview of the raw materials for pigment concentrates, including how to formulate and test them, and how additives by BYK can help you to get the best performance in the designated applications.

Note

To ensure the best appearance and full functionality, please open in Adobe Acrobat.

# **Definition of terms**

**Tinting pastes** are pigment concentrates used to adjust the color/shade of an already completed paint formulation. As usually small amounts are being used, the possible impact on the quality of the final paint is relatively minor.

# Universal colorants are

mainly used in architectural coatings to tint solvent- and water-based systems by using a single colorant series. A **full mixing system** is based on pigment concentrates suitable for producing complete paints by mixing with either clear or pigmented (mostly white or yellow) base paint. Higher amounts of pigment concentrates need to be added to the base paint, e.g. compared to tinting pastes. Therefore, the impact of those pigment concentrates on the final paint properties can be fairly significant.

# Overview of pigment concentrates used in architectural coatings





# Overview of pigment concentrates used in industrial coatings





# Advantages and challenges of pigment concentrates

Highly reliable and versatile coatings can be formulated by using pigment concentrates, and a variety of differently colored paints can be produced quickly, flexibly, and efficiently in short amounts of time and in small batch sizes.

However, to ensure the optimum performance, meaning the perfect balance between color variation and durability, it is vital that the pigment particles in the concentrate are deflocculated and well stabilized.

Storage stability

Achieving optimal pigment concentration of

(high-quality) pigments

Reproducible color in hue and color strength

Suitable rheological flow behavior (pourable and flowable without settling and syneresis) The use of wetting and dispersing additives, rheology additives, and defoamers by BYK ensures:

Compatibility

# **Selection of raw materials**

All raw materials used in the production of pigment concentrates have to:

# Meet the requirement of the highest quality paint system

Negative influence on durability in highly durable paint systems will not be accepted; therefore, the selection of the raw materials needs special attention. For example, if the cross linking in a 2-pack PU system is reduced, the hardness and durability is influenced.

# Be compatible with all let-down systems

What is important is the compatibility between:

- Wetting and dispersing additives and grinding resin;
- Wetting and dispersing additives and let-down systems (all let-down systems);
- Grinding resin and let-down systems (all let-down systems);
- Solvents and grinding resin;
- Solvents and let-down systems (all let-down systems)

### Meet regulatory requirements



# Solvent (or water)

The optimal solvent or solvent composition is related to the systems used and the solubility of the grinding resin. It should balance the compatibility with all systems and viscosity reduction in the pigment concentrate.

### Example:

In a solvent-borne 2-pack PU system, alcohols will have an influence on the crosslinking, while white spirit is not compatible. The use of esters, ketones, and aromatic solvents would be possible.

# **Grinding resin**

For solvent-borne pigment concentrates, grinding resins based on acrylates, aldehydes, ketones, polyesters, and alkyds are commonly used. Special attention is required in terms of the possible negative impact of the grinding resin on film properties of the final coating.

Grinding resins in water-based systems show a stronger limitation if compared to solvent-borne systems. One note for the water-based grinding resins: It is of the utmost importance that only shear stable resins are used.

# Optimum grinding resin level:

The optimum resin amount is a balance between pigmentation, rheology, and influence on the final paint properties. Generally, the amount of resin in solvent-borne pigment concentrates has an effect on viscosity, assuming that pigments are deflocculated and stabilized by wetting and dispersing additives.

# Influence of grinding resin amount in solvent-borne pigment concentrates

	Grinding resin amount	
Pigment load	Low	High
Viscosity	Increased	Decreased
Paint properties (durability, etc.)	High impact	Low impact
Thixotropic effect with organic pigments	Reduced thixotropic flow behavior	Increased thixotropic flow behavior
Correct balance will lead to a good flow behavior and stabilization of the pigment in the concentrates		

# Pre-test 1: Grinding resin and let-down systems

In order to evaluate the compatibility, 10% of the grinding resin as delivered should be added to the various nonpigmented let-down systems. There should be no turbidity, gelation (strong increase in viscosity) or precipitation visible right after incorporation as well as after 24-hours storage time at room temperature.

Also of importance is the compatibility of resins with the deflocculating wetting and dispersing additive, therefore a compatibility test should also be performed.

# Pre-test 1: Examples for incompatibility



# Pigment

The pigments have to cover the complete color range of the product portfolio to allow the mixing of all colors needed. They have to meet the requirements of the highest quality system in the range of let-down systems being used. Also of importance is the stability of the pigment in the media used (e.g. solvents, let-down systems, etc). If the pigment shows limited solvent stability (solvent fastness) the usage should be checked. Only if the pigment is stable in the solvents used in the pigment concentrates and let-down systems is it safe to use. Otherwise there could be problems regarding color stability or viscosity. Besides solvent stability (solvent fastness), the pH stability is also important, especially for water-based systems.

Information such as solvent fastness, light fastness, and area of usage normally can be found in the technical data sheets of the pigment. If solvent fastness is not mentioned, a simple test can help.

### Pre-test 2a: Solvent fastness

The pigment PY 74 can be used in systems which contain mineral spirit (white spirit) as solvent, but cannot be used in systems where aromatic solvents are used (for example: 2-pack PU or baking systems). The pigment PY 155 would be stable in both solvents. As such, it is the better choice for pigment concentrates.

# **Pre-test 2a:** Solvent fastness of two organic yellow pigments in two different solvents



No or slight coloration of liquid shows bleeding stability: pigment is stable and safe to use
 Strong coloration of liquid shows bleeding: pigment is unstable and inappropriate to use

G.06

# Pre-test 2b: Solvent fastness of different pigments in one solvent



# Wetting and dispersing additives

BYK's portfolio includes controlled flocculating and deflocculating wetting and dispersing additives. What are the differences and where is the area of usage?

Best optical properties for top coats including pigment concentrates will be achieved by deflocculating wetting and dispersing additives.

# Comparison of stabilization mechanisms



# Comparison of different stabilization mechanisms with regard to viscosity, settling, and syneresis



Settling tendency without anti-settling additive

# Importance of deflocculation

Primary particles (deflocculation) will show the original nature of the pigments:

- Increased gloss and reduced haze
- Reduced viscosity
- Prevention of seeding
- Selected color/hue
- Hiding power in case of opaque pigments or transparency, if nature of pigment is transparent

The selection of a deflocculating wetting and dispersing additive for pigment concentrates depends on:

- The polarity of the grinding resin
- The polarity of the let-down systems
  - Solvent-borne
  - Water-based
  - Universal usage
- Chemical nature of the pigment

# Pre-test 3: Compatibility of wetting and dispersing additives in grinding resin and let-down systems

In order to evaluate the compatibility, 5 % active substance of deflocculating wetting and dispersing additive should be added to the grinding resin and also to the various non-pigmented let-down systems. There should be no turbidity, gelation (strong viscosity increase), or precipitation visible immediately after incorporation as well as after 24-hours storage time at room temperature.

# Example: Deflocculated pigment red 101 (red iron oxide)

# Opaque



# Transparent



# **Rheology additives**

One requirement of a pigment concentrate is long-term storage stability. This is where rheology additives have a significant impact. They can be used as an anti-settling agent or to adjust the final viscosity. Especially deflocculated high-density (inorganic) pigments or fillers will create a strong tendency to settle. Rheology additives to prevent settling need to be formulated.

# Note:

Resin-free water-based pigment concentrates, even with a high pigmentation, often show low viscosity. Where tinting systems are concerned, this will be problematic if light colors have to be tinted. If lower pigmentation is required, a rheology additive will help to adjust the viscosity.

Rheology additives, e.g. based on polyurea (product codes: RHEOBYK-D 4xx or RHEOBYK-74xx) or on phyllosilicates, help to adjust the viscosity in various systems.

Particularly for universal colorants including thickeners, it is of upmost importance to evaluate the interdependency on water-based as well as on solvent-borne systems.

# **Rheology additives for pigment concentrates**

	Solvent	Water	Universal
GARAMITE Organophillic phyllosilicates	•		
RHEOBYK-D 4xx and RHEOBYK-74xx Polyurea	•	•	•
LAPONITE Synthetic phyllosilicates		•	
OPTIGEL Modified phyllosilicates		•	•
BYK-AQUAGEL Modified phyllosilicates		•	•

# Influence of rheology additives on colorant acceptance



If the defoamer, which is used in the pigment concentrate, is too incompatible, it might affect the gloss, haze, or disturb the leveling (e.g. tendency to crater).

### Defoamer

Foam formation during the grinding process influences the grinding efficiency. Therefore, especially water-based pigment concentrates need a defoamer with a good balance between efficiency and compatibility. During the complete grinding process, the additive should de-aerate and defoam the pigment concentrate. During the final application, the defoamer should not influence the paint performance and paint quality. If the defoamer, which is used in the pigment concentrate, is too incompatible, it might affect the gloss, haze, or disturb the leveling (e.g. tendency to crater).

In solvent-borne pigment concentrates, foam tendency is not that strong, so defoamers are rarely used.

Water-based pigment concentrates (either only for waterbased systems or as universal colorants) need a defoamer during the grinding phase. In the coating industry, the trend towards environmentally-friendly systems pushes the formulator to use mineral-oil-free defoamers such as silicone or polymeric defoamers.

For some formulations with a special purpose, the addition of a defoamer after the milling process is required. It is not necessarily important to use the same defoamer as used during milling.

# Formulating guideline

The correct combination of pigment binder ratio and additive dosage will provide a good balance between pigmentation, viscosity, stabilization, and storage stability. The following pages show some examples with different grinding resins and different usage areas.

# Architectural coatings (1/2)

# Example: Solvent-borne pigment concentrates based on high solid alkyd resin

# Example: Solvent-borne pigment concentrates based on alkyd resin

	Pigment : binder ratio (solids : solids)	Additive dosage in % (solids on pigment)
Titanium dioxide	2.0-2.5 : 1	2–3
Organic yellow	0.4-0.5 : 1	15–25
Inorganic yellow	0.8-1.0:1	7–9
Organic orange	1.0-1.2 : 1	18-25
Organic red	0.4-1.0:1	18-23
Inorganic red	2.5-2.8 : 1	8–12
Organic red violet (magenta)	0.2-0.4:1	25-35
Organic violet	0.2-0.4:1	30-40
Phthalo blue	0.2-0.3 : 1	20-30
Phthalo green	0.2-0.3 : 1	25-35
Coarser carbon black (e.g. Lamp black 101)	0.3-0.4:1	25-40
Carbon black (e.g. Special black 4)	0.3-0.4:1	30-50

(solids : solids)	(solids on pigment)
4.0-5.0:1	2-3
3.0-3.5 : 1	15-25
4.0-5.0:1	7-9
1.0-1.5 : 1	18-25
1.5-2.0 : 1	18-23
4.5-5.0:1	8-12
1.0-1.5 : 1	25-35
1.0-1.5 : 1	30-40
0.9–1.3 : 1	20-30
0.9–1.3 : 1	25-35
0.7-1.0:1	25-40
0.7-1.0:1	30-50
	(solids : solids)         4.0-5.0 : 1         3.0-3.5 : 1         4.0-5.0 : 1         1.0-1.5 : 1         1.5-2.0 : 1         4.5-5.0 : 1         1.0-1.5 : 1         1.0-1.5 : 1         0.9-1.3 : 1         0.9-1.3 : 1         0.7-1.0 : 1

# Architectural coatings (2/2)

T. 04

# Example: Water-based, resin-free pigment concentrates

# Example: Water-based, resin-free universal colorants (solvent- and water-based applications)

	Additive dosage in % (solids on pigment)
Titanium dioxide	1–3
Organic yellow	15–25
Inorganic yellow	7–9
Inorganic yellow transparent	20-25
Organic orange	15–20
Organic red	17–20
Inorganic red	5-7
Inorganic red transparent	25-28
Organic red violet (magenta)	30-35
Organic violet	30-35
Phthalo blue	20-30
Phthalo green	25-30
Coarse carbon black (e.g. Lamp black 101)	35-45
Medium carbon black (e.g. Special black 4)	40-50

	Additive dosage in % (solids on pigment)
Titanium dioxide	2-5
Organic yellow	10–15
Inorganic yellow	6-7
Organic orange	15–20
Organic red	15–20
Inorganic red	5-7
Organic red violet (magenta)	25-30
Organic violet	35-40
Phthalo blue	25-35
Phthalo green	15–25
Coarse carbon black (e.g. Lamp black 101)	35-40
Medium carbon black (e.g. Special black 4)	30-40

T. 05

# Industrial coatings

# Example: Solvent-borne pigment concentrates based on aldehyde resin

Example: Water-based pigment concentrates (resin-free or resin-containing)

	Pigment : binder ratio (solids : solids)	Additive dosage in % (solids on pigment)
Titanium dioxide	4.5-5.0 : 1	2-4
Titanium dioxide, transparent	1.0-2.0 : 1	15-20
Organic yellow	3.0-3.5 : 1	15-20
Inorganic yellow	4.0-5.0:1	7–10
Inorganic yellow, transparent	1.0-2.0 : 1	15-20
Organic orange	1.0-2.5 : 1	15-35
Organic red	1.0-2.5 : 1	20-30
Inorganic red	4.0-5.0:1	8-12
Inorganic red, transparent	1.0-2.0 : 1	15-20
Organic red violet (magenta)	1.0-1.2 : 1	15-45
Organic violet	1.0-1.2 : 1	25-40
Phthalo blue	0.9–1.3 : 1	20-40
Phthalo green	0.9–1.3 : 1	20-35
Coarser carbon black (e.g. Lamp black 101)	0.5-1.0:1	15-30
Carbon black (e.g. Special black 4)	0.5-1.0:1	30-50
Fine carbon black (e.g. FW 200, Raven 5000 Ultra)	0.3-0.5 : 1	50 – 90

	Additive dosage in % (solids on pigment)
Titanium dioxide	3–5
Titanium dioxide, transparent	20–25
Organic yellow	15-20
Inorganic yellow	7–10
Inorganic yellow, transparent	20–25
Organic orange	15-30
Organic red	15-30
Inorganic red	7–12
Inorganic red, transparent	20-25
Organic red violet (magenta)	25–35
Organic violet	25-35
Phthalo blue	20-40
Phthalo green	25–35
Coarse carbon black (e.g. Lamp black 101)	30-40
Medium carbon black (e.g. Special black 4)	50-70
Fine carbon black (e.g. FW 200, Raven 5000 Ultra)	60-90

T.06 Note: Resin-containing water-based concentrates include around 5–15% solid resin

T. 07

# Preparation of pigment concentrates in lab scale

- **1.** Premix grinding resin (solution), solvent/water and wetting and dispersing additive
- 2. Add rheological modifier/anti-settling additive, if needed
- 3. Add pigment, pre disperse for 10–15 minutes
- 4. Perform grinding
  - **a.** Inorganic pigments: high-speed mixer, bead, pearl, or sand mill
  - b. Organic pigments: bead, pearl, or sand mill

It is important for grinding that the mill base shows the so-called vortex effect (doughnut effect) during the complete grinding process.

BYK starting formulations are tested on our lab scale grinding equipment. Various grinding equipment is available on the market for lab scale or production. Therefore, the formulations must be adapted to the specific grinding equipment and let-down system.

**Vortex effect** 

(doughnut effect)

An animation of the vortex effect

can be found online.

### Important

A uniform flow of the mill base which will be ground should be ensured during the entire dispersing process.

G. 12

# Testing of pigment concentrates

The success of a sufficient dispersion can be checked via grinding gauges (Hegman gauges), as this test indicates the presence or absence of oversized agglomerates and thus improper dispersion. However, this test method does not indicate whether the pigments are properly deflocculated and stabilized against re-agglomeration, as the primary particles of organic pigments or inorganic pigments such as iron oxide red are way below 1 µm and cannot be seen in a test with a grinding gauge. There are several test methods that indicate sufficient dispersion and stabilization of a pigment concentrate by testing them within a coating system and measuring various properties.

# Possible tests are:

- Viscosity measurement
- Color/hue evaluation
  - Color strength
  - Determination of color shift/visual assessment, page 22
  - Rub-out test for color stability (flooding and floating), page 23
  - Colorant acceptance, page 25

# Physical properties:

- Gloss
- Haze
- Seeding
- Durability test
- Weather resistance
- Substrate adhesion or intercoat adhesion
- etc.
- Particle size, page 26
- Storage stability, page 27



# Determination of color shift/visual assessment

In full shades, where a pigment concentrate is incorporated into a clear coat formulation, a visual assessment can be performed to indicate if the pigments are properly dispersed and stabilized.

For transparent pigments, their state of deflocculation is indicated by the transparency of the coating. When the pigments are flocculated, they scatter the transmitted light which results in a visible turbidity. Additionally, the color shift visible in the comparison of two samples can indicate whether or not the pigments are stabilized. If pigments are flocculated, the light is reflected with a slightly different wavelength compared to deflocculated pigments. This leads to a color shift/shift in hue between the samples. To determine the stabilization, a color diagram can be used. It is read in the following way: green pigments appear more bluish when they are deflocculated and more yellowish if they are flocculated, red pigments appear more blueish when flocculated and more yellowish when deflocculated.

# Color shift of different pigments caused by flocculation/deflocculation



Deflocculation of a red pigment shows a color shift in the direction of yellow.

Flocculated

Deflocculated

In transparency, deflocculation of a green pigment shows a color shift towards blue, in addition to an improvement in transparency.

 $\equiv$ 

### **Rub-out test**

The basic principle behind the rub-out test is to check whether or not the pigment is properly stabilized. The pigment concentrate is not tested directly, but incorporated into a let-down system to obtain either a full-shade system (one single pigment) or a color blend system (containing the pigment concentrate to be tested and a stable second pigment, such as titanium dioxide). The paint is applied and allowed to dry. During drying, the improperly stabilized

pigments agglomerate, which leads to a change in hue. Before the paint dries completely, the surface is rubbed with a finger, either from side to side or in a circle, which redisperses the pigments with the shear force of the rubbing (see G. 14). This can lead to a significant change in color compared to the unrubbed area, and indicates the reagglomeration of the pigment.

The correct time to perform the test is when the paint is almost dry but still tacky. If your finger leaves marks on the paint surface, it is an indication that this is the right moment to conduct the rub-out test.

# Full shade of opaque PR 101 with rub-out test



### Due to the uneven surface at the rubbed area, it is recommended to use color measurement equipment with sphere geometry (d8°).

### **Rub-out test in color blends**

If all full shades in every let-down system show good performance, a color blend should be tested. As a first step, one color blend with white is recommended for each system.

# Example:

- Let-down system 1
- + Titanium dioxide concentrate
- + Iron oxide red concentrate
- Let-down system 1 + Titanium dioxide
- concentrate
- + Phthalo green
- concentrate • Let-down system 1
- + Titanium dioxide
- concentrate
- + ... concentrate

- Let-down system 2 + Titanium dioxide
- concentrate + Iron oxide red concentrate
- Let-down system 2 + Titanium dioxide concentrate
- + ... concentrate

and so on.

• Let-down system 3 + ...

Only if there is no color difference ( $\Delta E$  depends on requirements), an additional test, such as a storage test, should be performed. If one or more system/concentrate shows a strong color difference, an improvement in results is needed and a cross-check in every system/color must be carried out.

Therefore, it is recommended to start only with titanium dioxide concentrate and three additional colored pigment concentrates (e.g. iron oxide red, organic pigment, and carbon black).

For all these mixtures (white reduction or white mixtures),

a rub-out test after the application should be performed.

# White reduction PR 101 pigment concentrate in one let-down system

# White reduction 1

# PW 6: Insufficiently stabilized PW 6: Insufficiently stabilized PW 6: Stabilized PW 6: Stabilized PK 101: Insufficiently stabilized PK 101: Stabilized PK 101: Stabilized PK 101: Stabilized

# White reduction 2

G. 15

# **Colorant acceptance**

The stabilization of pigments and the compatibility of the colorant in the base paint can be determined by the colorant acceptance test.

In this test, two samples are compared with each other. For the first sample, the colorant is incorporated into the base paint at low shear forces (e.g. stirring by hand), for the second sample, it is incorporated at significantly higher shear forces, e.g. using high-speed dispersion equipment or a paint shaker. Both paints are applied onto a suitable substrate, and a rub-out test is carried out. If the colorant is sufficiently stabilized and compatible, the rub-out test should show a good result in both samples. If a difference can be seen between the high shear force and the low shear force sample, the colorant should be checked for stability or compatibility with the base paint.

or

# Incorporation of pigment concentrates into the base coat with different shear forces



High shear force



Paint shaker



High-speed dispersing

# Particle size

The state of flocculation/deflocculation and the resulting particle size can be directly observed using a microscope. However, this observation cannot be carried out directly with the pigment concentrate. It needs to be incorporated into the base paint first, and the base paint then diluted with two to four parts of the solvent that is used in the paint system. Depending on the size of the agglomerates, it can be checked whether the pigment particles are flocculated or deflocculated.



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# Storage stability

Pigment concentrate will not be used up completely after production, therefore it is important that the concentrates show good results both before and after storage. It is recommended to test stored pigment concentrates in the same way as conducted during the evaluation (full shades: color drift, gloss haze, seeding, and white reductions/white mixtures: rub-out, color strength, gloss, haze, and colorant acceptance).

# Storage stability tests – Determination of required actions

Results of lab scale		Action
Before storage	After storage	
Good	Bad	Adjust the pigment concentrate formulation
Good	Good	<ul> <li>Start pilot plant production</li> <li>Evaluate mixing ratios for required mixing colors</li> </ul>





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