

Dispersing Technology

EDAPLAN[®] and METOLAT[®]

1 Introduction	Page	1
2 Basics of pigment grinding	Page	3
3 Dispersing process	Page	5
4 Different mechanisms of wetting and dispersing agents	Page	6
5 Structure of wetting and dispersing agents	Page	7
6 Use of EDAPLAN as unique dispersing and wetting additive in the grinding	Page	9
7 Determination of the optimum dispersant demand	Page	10
8 Test methods to check the quality of a pigment dispersion	Page	12

1. Introduction

For the production of water based paints and coatings two different types of formulations can be used. The matter of differentiation is the stability of the binder to shear forces and temperature.

a) *Use of water soluble resins as binder or dispersing resin:*

The resins are shear and temperature stable \Rightarrow the use in the mill base is possible.

Depending on the chemical composition of the resins and the type of pigments a dispersant is not necessary.

b) *Use of polymer emulsions:*

Emulsions normally are not shear and temperature stable \Rightarrow the use in the mill base is not possible. A dispersant has to be added to the mill base.

If it is not possible to use the binder in the mill base, the dispersion of the pigments has to be done by dispersing agents in a previous step. This has led to the development of (universal) pigment pastes. These pigment paste systems are used in the production of water based industrial and automotive paints and architectural coatings for color matching.

In a next development step semi-finished products are used to produce paints in order to simplify and rationalize production processes.

The components of such production systems are for example:

- pigment paste systems (tinters)
- extender containing basic paints
- clear coatings

In order to achieve a rationalization by such a method, the pigment pastes have to be as compatible as possible with a wide range of paint systems.

The following profile of requirements for such a tinting system can be created. Some of these requirements are also valid for mill base formulations.

- *Storage stability:*
Stable to sedimentation and flocculation (color strength has to maintain constant)
- *Flow characteristics:*
Pumpable, usable with automatic dosing systems
- *Compatibility with coating systems and binders:*
Chemical and physical properties and resistances may not be influenced negatively. The processing properties and the storage stability of the coatings have to be preserved.
- *Economy:*
High tinting strength.
High pigment content and/or standardized tinting strength in all colors of a tinting system.

In order to achieve the above mentioned requirements, special properties of the pigments pastes are necessary. These properties are obtained by dispersing additives.

The requirements for dispersing agents are as follows:

- Support of grinding ⇒ short dispersing time, optimum degree of grinding.
- Reduction of grind viscosity ⇒ high pigment content, flowability.
- Stabilization of the pigments ⇒ color strength, gloss, color shade stability.
- Compatibility with resins ⇒ no flocculation or coagulation, broad application.

2. Basics of pigment grinding

Pigments

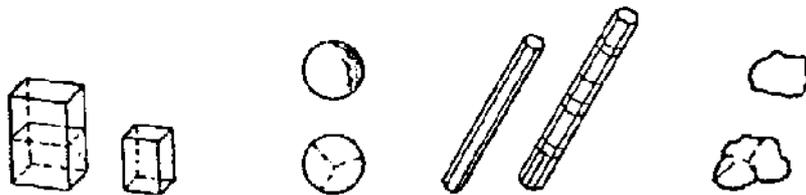
A pigment is a coloring particle, which is insoluble in the application media. They have different particle forms and sizes and are based on chemically completely different structures. The optimum size of the particles in order to obtain certain application properties like gloss, tinting strength and weather fastness is in the range of the wavelengths of visible light.

In powder form the pigments are found in a particle size distribution, which is caused by the production processes. The particle sizes vary around an average. Additionally, pigments are linked together by agglomeration to bigger particles. The particle size of commercially available pigments is much higher than the ideal size.

- **Primary particles**

Size and crystal structures are influenced during the production process.

Primary particles are single crystallites or sub crystallites, which are strongly connected by their surface areas. They can't be destroyed during normal grinding processes.

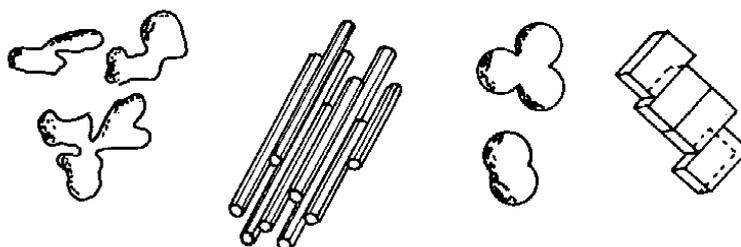


- **Aggregates**

Size and form can be influenced during the production process of the pigments.

Aggregates are particles, in which primary particles are connected together by their surfaces. As a result, the total surface of aggregates is smaller than the sum of the surface of the primary particles in the aggregate.

Aggregates are as difficult to destroy as primary particles in the grinding process.

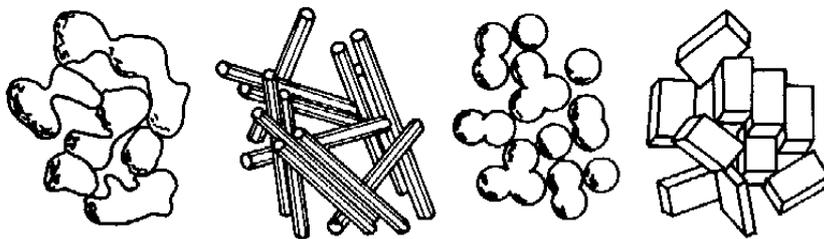


- **Agglomerates**

Agglomerates are formed in a pigment powder or in pigment preparations, which are not perfectly stabilized.

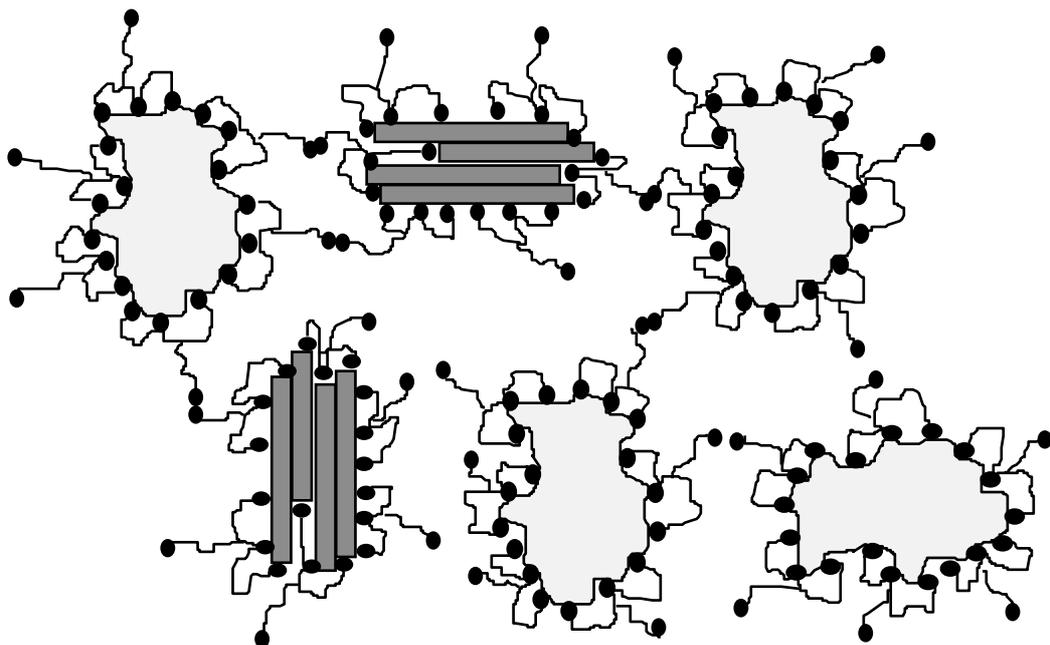
Agglomerates are an easy connected composite of primary particles and aggregates. They are only connected with edges and borders and therefore their total surface area is nearly identical with the surface of the primary particles and aggregates in an agglomerate.

Agglomerates can have particle sizes up to several 100 µm. The dispersion of agglomerates is the main process in the grinding process.



- **Flocculates**

Flocculates are formed by particles, which are already dispersed or by particles, which are wetted by binders or additives. Binders or additives, which are adsorbed on the pigment surfaces, can cause interactions between the particles. Flocculation can be useful against floating and flooding effects, but they can exhibit also negative effects on gloss and color shade.



3. Dispersing process

There are three different steps in the dispersion process, which take place at the same time and/or after each other:

1. *Wetting of pigment surfaces*

- The air or water covering a pigment surface is substituted by binders or wetting and dispersing agents.

2. *Destroying of agglomerates*

- Acting of mechanical forces:
Shear forces, which are present in the liquid phase of the grinding.
- physical and chemical interactions:
Wetting and entering of the liquid components into pores of agglomerates. The adhesion forces between pigment particles are substituted by interaction forces of liquid molecules (binder molecules, additives, solvents). These forces are a power of ten lower than the adhesion forces between the particles. Due to that, agglomerates can be easily destroyed or are even broken by the liquid (instant effect).

3. *Stabilization of a dispersion*

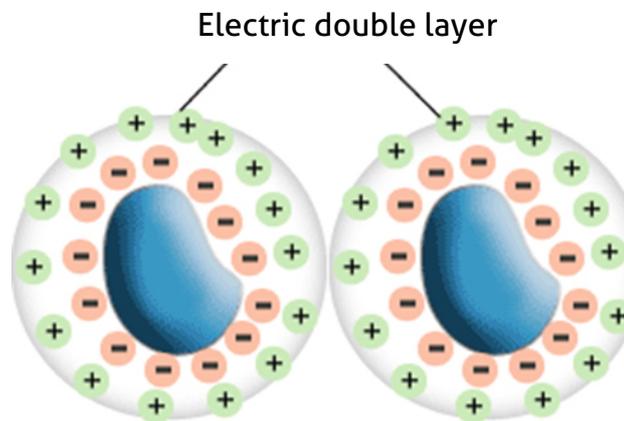
- Prevention of reagglomeration by dispersing additives
- Prevention of flocculation

4. Different mechanisms of wetting and dispersing agents

In the literature generally two principally different mechanisms for the stabilization of dispersions are described: electrostatic and steric stabilization.

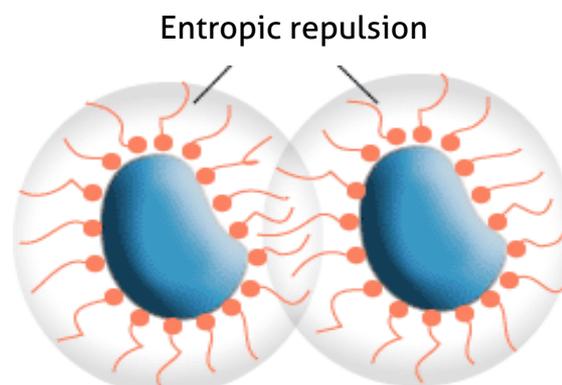
a) *Electrostatic stabilization*

The surface of the pigments is occupied by additives, which are ionically charged and an electric double layer is build up. All pigment particles are charged in the same way. The mutual repulsion due to the charges is bigger than the attraction forces between the pigments.



b) *Steric stabilization*

Pigment affine groups accumulate on the pigment surface. The molecular chains widely extend into the surrounding media and need a certain space in order to be able to move freely. During the approach of pigment particles, the mobility of the polymer chains is reduced which leads to a loss of entropy. This leads to a repulsion force. The hydrophobic parts of polymer show an extremely long and lasting interaction with pigment surfaces.

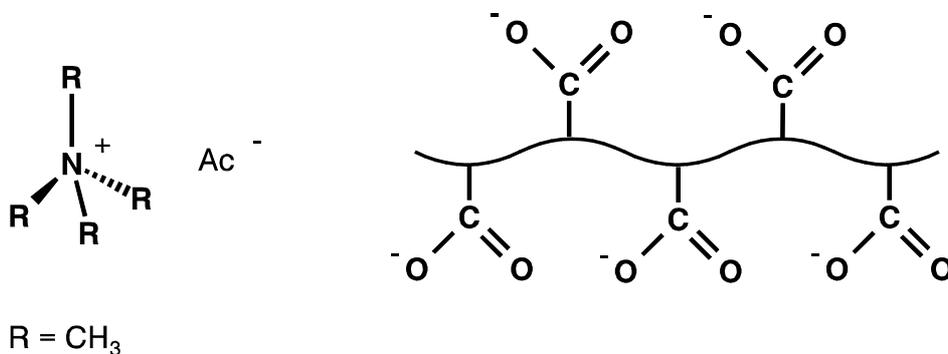


5. Structures of wetting and dispersing agents

a) Electrostatically acting dispersing agents

These products are dispersing agents containing cationic or anionic molecule groups. For example quaternary ammonia salts and alkylpolyamines (cationic) or polycarboxylic acids and sulfonated organic substances (anionic).

From the range of dispersing agents of MÜNZING CHEMIE GMBH: **METOLAT® 514**
EDAPLAN® 516



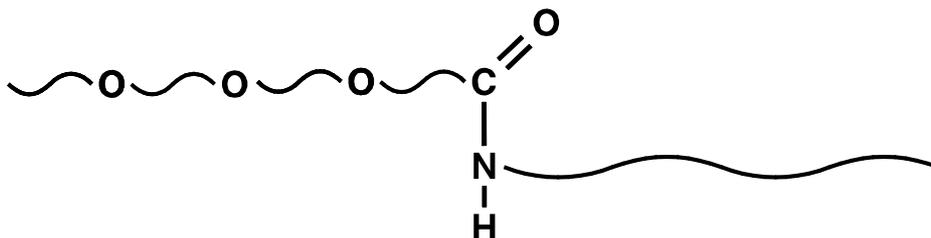
b) Sterically acting dispersing agents

These products are nonionic or mainly nonionic substances.

Hydrophilic groups (e.g. polyether) and pigment affine groups (e.g. amino groups) containing oligomers or polymers.

From the range of wetting and dispersing agents of MÜNZING CHEMIE GMBH:

for organic pigments **METOLAT® 355**
METOLAT® 388

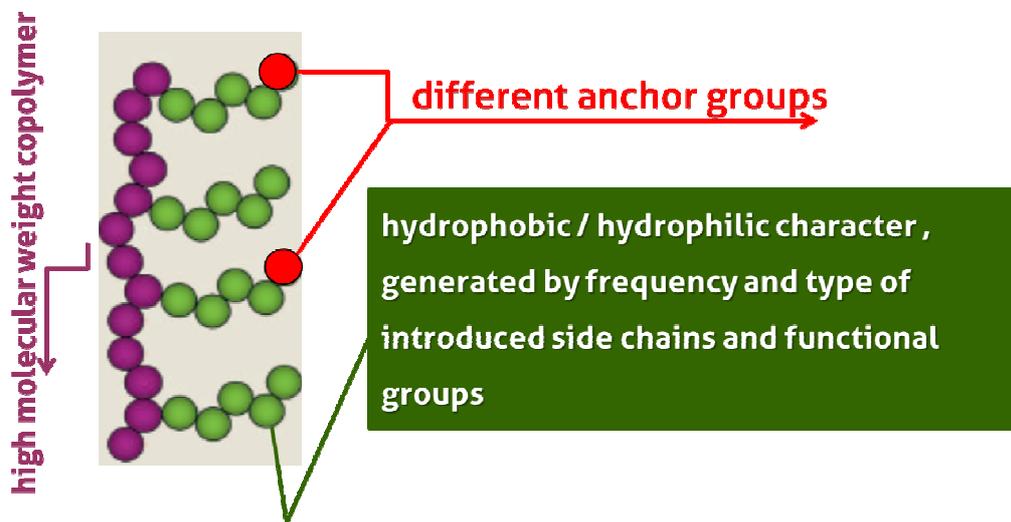


c) *Combination of sterically and electrostatically acting dispersants*

Polymeric dispersing agents unify the properties of classical wetting and dispersing agents. The backbones carry the charges and at the same time are modified with surface active groups. Pigment affine molecule parts increase the adsorption onto the pigment surface and therefore rise efficiency.

From the range of polymeric dispersing agents of MÜNZING CHEMIE GMBH:

EDAPLAN® 490 series



Dosage	
in pigment pastes	0.5 - 10 % inorganic pigments
	5 - 30 % organic pigments
	20 - 60 % carbon black
in direct grinding	0.5 - 10 % inorganic pigments
	5 - 30 % organic pigments
as compatibility agent	approx. 0.1 % on white paints

6. Use of EDAPLAN® polymeric dispersants as sole dispersing and wetting additive in the grinding

The polymeric dispersing agents EDAPLAN® 490, 492 und 494 can substitute common dispersing and wetting agents, which are used in the grinding process of paint production. In the normal grinding process of paints and lacquers dispersants like polyacrylates and polyphosphates are used. For the wetting of organic pigments further ionic or nonionic wetting agents are added. Dosages in the sum of all additives might be high due to the limited performance of these products.

The polymeric dispersants EDAPLAN® act as a combination of wetting and dispersing agent and can substitute these products efficiently. Instead of using two or three products the formulation can be reduced to only one product. Apart from the high compatibility of the EDAPLAN® products the high efficiency of dispersing has to be pointed out. EDAPLAN® polymeric dispersing agents show a very high compatibility with common binder systems and different types of pigment pastes.

7. Determination of the optimum dispersant demand

All pigments, which are used in paints and coatings systems have different particle sizes and specific surfaces due to their chemical structure and in order to achieve the desired optical and application properties. Therefore different dispersant types and various amounts are needed in order to guarantee a complete covering of the pigment surface with the dispersing agent.

The complete covering of the surface is one of the basic requirements to achieve an ideal stabilization of the dispersed pigments.

In aqueous pigment slurries the required amount of dispersing agent is relatively easy to determine in a first oriented step. The fact, that the viscosity of a pigment slurry reaches a minimum, when the pigment surface is completely covered with a dispersant is used to determine the dispersant demand.

An often used test method is described as follows:

7.1 Method A

1. Preparation of a pigment slurry:

The pigment content is chosen such that after dispersion a non flowable pigment paste is generated. Normally a content of approximately 30% organic pigment and 70% inorganic pigment is necessary to obtain these conditions.

The dispersing time is about 30 min.; a dissolver or an equal dispersing device is used.

2. Addition of dispersant/determination of viscosity:

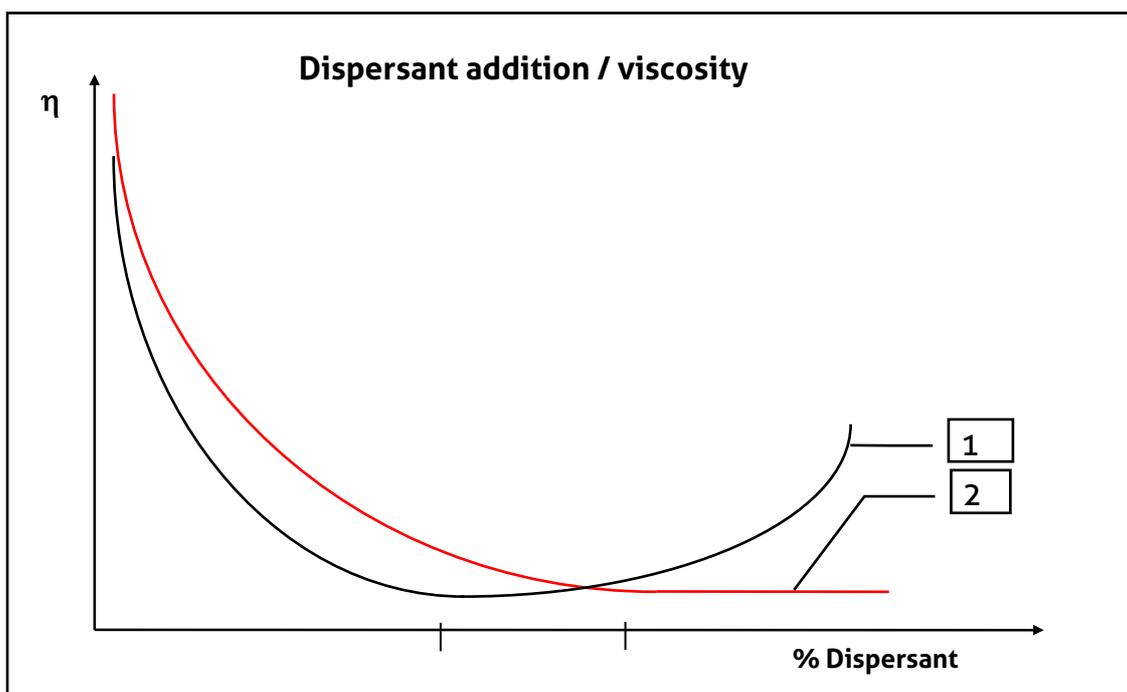
The dispersant is added in portions to the stirred pigment slurry. After the addition and mixing the viscosity is measured at low shear rates (e.g. with a Brookfield viscometer or a cone and plate system).

The dispersant is added until a viscosity minimum is obtained.

In this mode of determination two typical viscosity curves can be observed:

Graph [1] occurs more or less strongly with conventional wetting agents and dispersants for inorganic pigments like polyphosphates and polycarboxylic acids.

Curve [2] is typical for polymeric dispersing agents.



In general, the amount of dispersing agent, which is determined in the experimental method A, is too low. The reasons are:

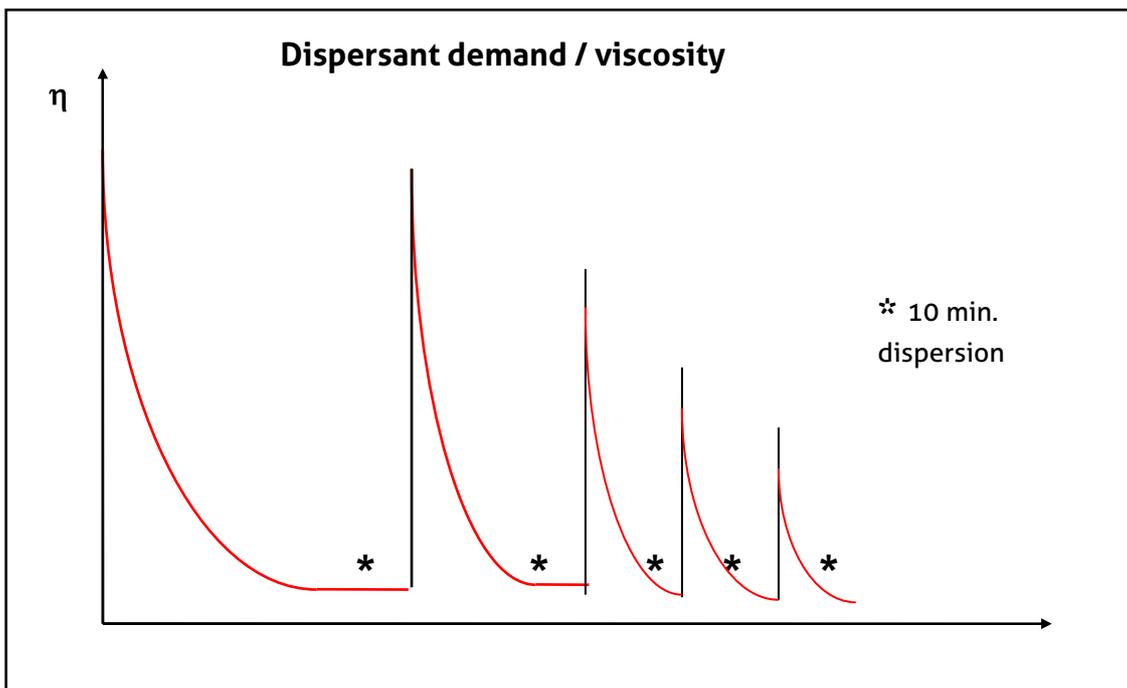
- the optimum degree of grinding is not achieved, because the amount of dispersing agent or binder during dispersion is too low (\Rightarrow basics of pigment grinding)
- reagglomeration starts immediately after dispersing process and the amount of dispersing additive or binder to stabilize the pigment particles is too low.

This can be demonstrated, if the procedure is changed to the method described in 7.2.

7.2 Method B

1. Dispersing of a pigment slurry with glass beads in a shaking mixer (e.g. „Red-Devil“)
2. Addition of dispersant until a minimum of viscosity is obtained. Dispersing is continued (often 10 minutes are sufficient). In many cases the viscosity rises again and more dispersing agent has to be added to the slurry. The procedure is continued until a clear minimum in viscosity is obtained.

The following graph shows the typically viscosity/dispersing demand curve of polymeric dispersants:



The viscosity values decrease after each addition of dispersant to a minimum, but rise again after a longer dispersing time. Viscosities can be lowered stepwise until no final increase in viscosity is observed any more. The quantities of dispersing agent found in this determination method, however, are not indicative for the obtained particle size, the stability against reagglomeration and the stability of the adsorbed dispersants on the pigment surface.

8. Test methods to check the quality of a pigment dispersion

In order to determine the quality of a dispersion by indication of the particle size, stability of dispersion and stability of adsorbed dispersants the following test method can be applied:

1. Determination of the dispersant demand:

The exact amount of dispersing agent is determined following method B (chapter 7.2).

2. Preparation of pigment pastes with the following formulation:

The pigment pastes are prepared with the dispersant quantity thus determined.

Additionally, pastes are prepared with a dispersant amount 10% less and 10% higher than the optimum value in order to prove the determined amount in a pigment paste preparation.

	organic pigments	inorganic pigments	carbon black
pigment	30 - 40%	60 - 70%	10 - 20%
dispersant	value determined		
neutralising agent	depending on amount dispersant (only EDAPLAN® 470 and 472)		
defoamer	1%	0,5%	1%
water	rest on 100 %		
	100%	100%	100%

Dispersing is performed at a shaking mixer („Red-Devil“ or „Scandex“; in order to obtain comparable values always the same device has to be used).

Ratio pigment paste to glass beads = 1: 1.5 % parts; dispersing time: 30 - 60 min.

After grinding clear coatings are pigmented with the prepared tinters and applied on glass plates.

Ratio pigment to binder for clear coatings (pigment : binder_{solid})

Inorganic pigments: 20 - 30 %

Organic pigments: 10 - 15%

Carbon black: 5 - 10%

3. Evaluation of a dispersion

The following test methods can be performed in order to determine and compare the quality of a dispersing agent and the dispersion.

Gloss, haze gloss measurement	⇒	Comparative evaluation of particle size
Color shade, color strength	⇒	Particle size, color changes caused by chemical reaction of dispersant and pigment. Changes of primary particle size or structure.
Rub-Out	⇒	Reagglomeration, flocculation, floating/flooding
Different application methods (e.g. rolling, spraying)	⇒	Flocculation, formation of agglomerates
Transparency or hiding power	⇒	Fineness of grinding
Viscosity measurements	⇒	Rheological profile, flow behavior