

Dispersing Technology

EDAPLAN[®] and METOLAT[®]

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1. Introduction

For the production of waterbased paints and lacquers in principle two different types of formulations for the fabrication can be used. The matter of differentiation in these cases 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 its use in a 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 its 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 reducible industrial and automotive paints and protective building coatings for colour matching and tint correction.

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

The components of such production systems are for example:

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

In order to achieve a rationalisation by such a method the tinters 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 developed. Some of these requirements are also valid for mill base formulations.

- *Storage stability:*
Stable to sedimentation and flocculation (colour strength has to maintain constant)
- *Flow characteristics:*
Pumpable, processible 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 maintain.
- *Economy:*
High tinting strength.
High pigment content and/or standardised tinting strength in all colours of a tinter system.

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

The requirements for dispersing agents therefore are as follows:

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

2. Basics of pigment grinding

Pigments

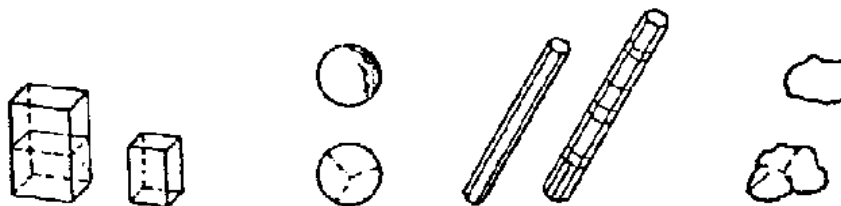
A pigment is a colouring particle which is insoluble in the application media. They appear in different particle forms and sizes and are based on chemically completely different structures. The optimum size of the particles in order to obtain certain application properties like gloss, tinting strength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

In a powderous form they are supplied in the pigments are found in a distribution of particle sizes which is caused by the production processes. The particle sizes vary around a mean. Additionally, pigments are linked together by agglomeration to bigger particles which in fact means that the particle size of commercially available pigments is much higher than the ideal size.

- **Primary particles**

Size and crystal structure are influenced during the production process.

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

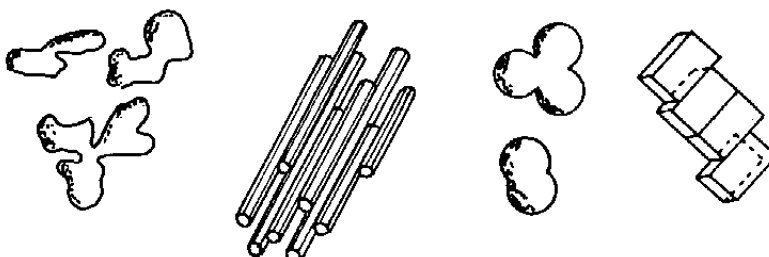


- **Aggregates**

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.

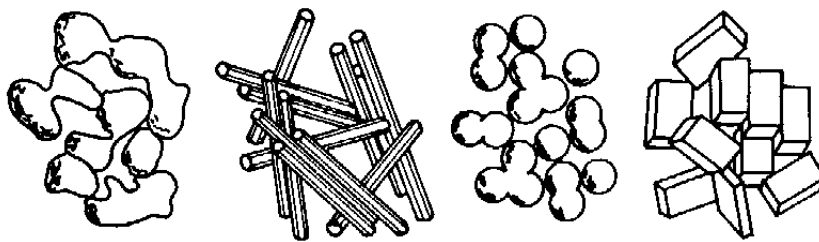


- **Agglomerates**

Agglomerates are formed in a pigment powder or in pigment preparations which are not stabilised enough.

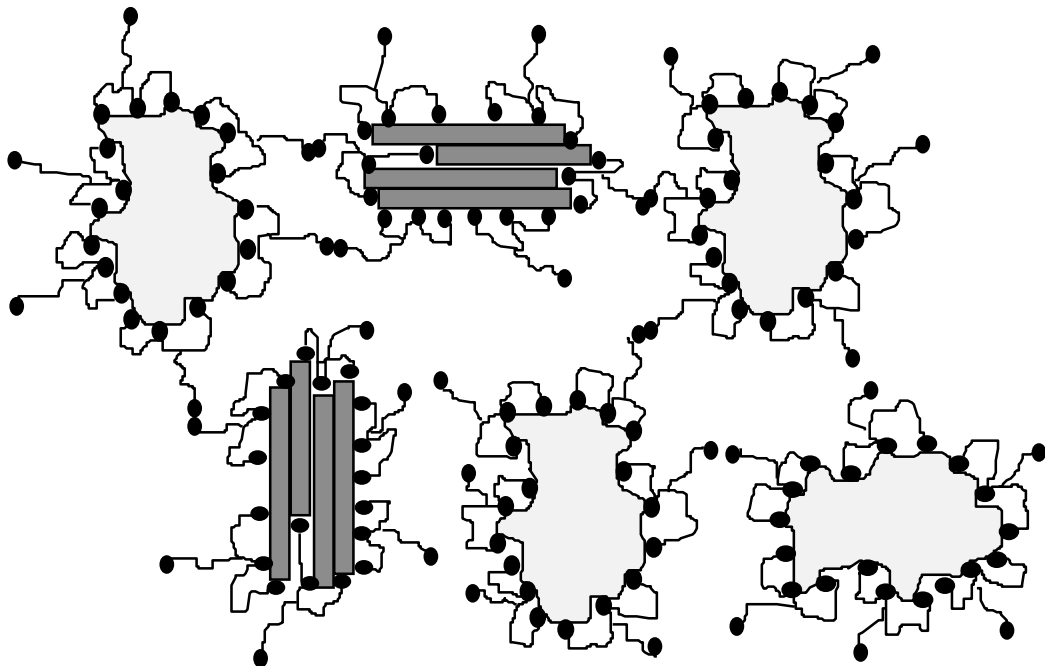
Agglomerates are an easy connected composite of primary particles and aggregates. They are only connected with edges and borders and therefore their total surface area is nearly identical with the surface of the primary particles and aggregates in an agglomerate.

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 dispersed already or by particles which are wetted by binders or additives. Binders or additives which are adsorbed on the pigments 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 colour shade.



3. Dispersing process

Following three processes are meant with the term dispersing at the production of coatings. These three steps take place at the same time and/or after each other:

1. *Destroying of agglomerates*

- Acting of mechanical forces:
Shear forces which are transferred mainly by the liquid phase of the grinding onto pigments
- 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 tenth power 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).

2. *Wetting of pigment surfaces*

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

3. *Stabilisation of a dispersion*

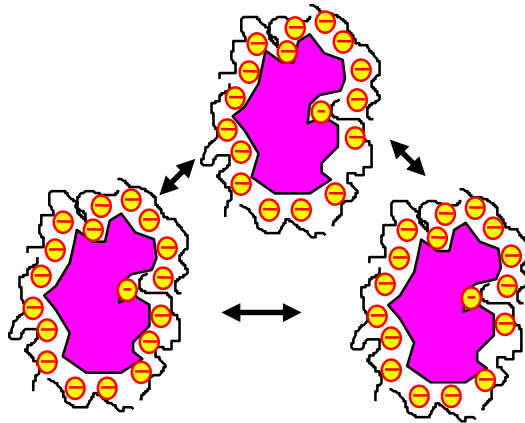
- Prevention of reagglomeration by dispersing additives
- Prevention of flocculation or admission of a defined flocculation

4. Different mechanisms of wetting and dispersing agents

In the literature generally, two principally different mechanism for the stabilisation of dispersions are described: electrostatical and sterical stabilisation.

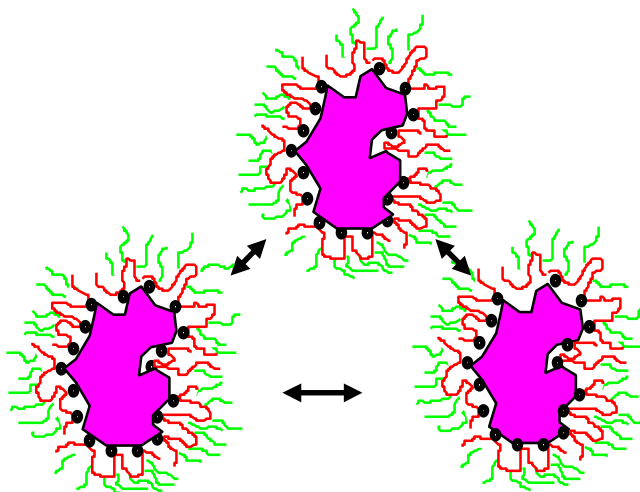
a) *electrostatical stabilisation*

The surface of the pigments is occupied by an additive which is ionically charged. All pigment particles are charged in the same way. The mutual repulsion due to the charges is bigger than the forces of attraction between the pigments.



b) *sterical stabilisation*

The attraction forces between the pigment particles are only effective over relatively short distances. The approach of two particles to each other is prevented by molecules which are absorbed on the pigment surface and which have groups which have a long chain ranging into the liquid phase. Agglomerations can be prevented because pigment particles can not get close to each other due to these long chain groups.

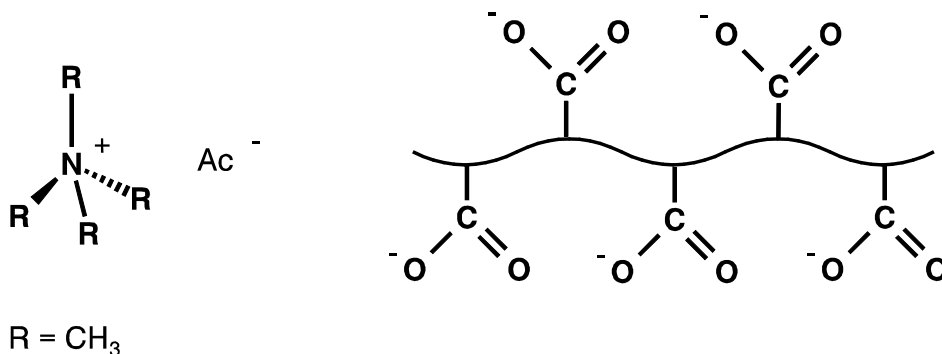


5. Structures of wetting and dispersing agents

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).

The range of dispersing agents from MÜNZING CHEMIE GMBH: **METOLAT® 514**



b) *sterically acting dispersing agents*

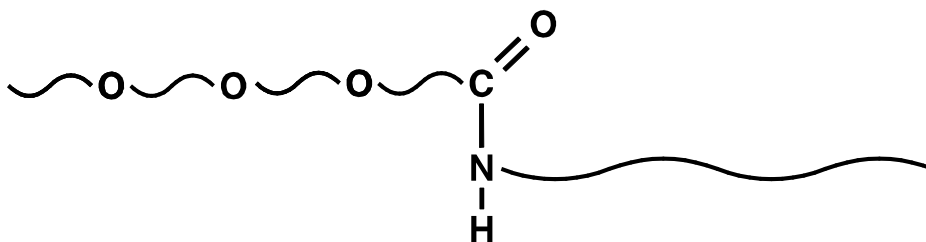
These products are nonionic or mainly nonionic substances.

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

The range of wetting and dispersing agents from MÜNZING CHEMIE GMBH:

for organic pigments and substrates **METOLAT® 355**

METOLAT® 388



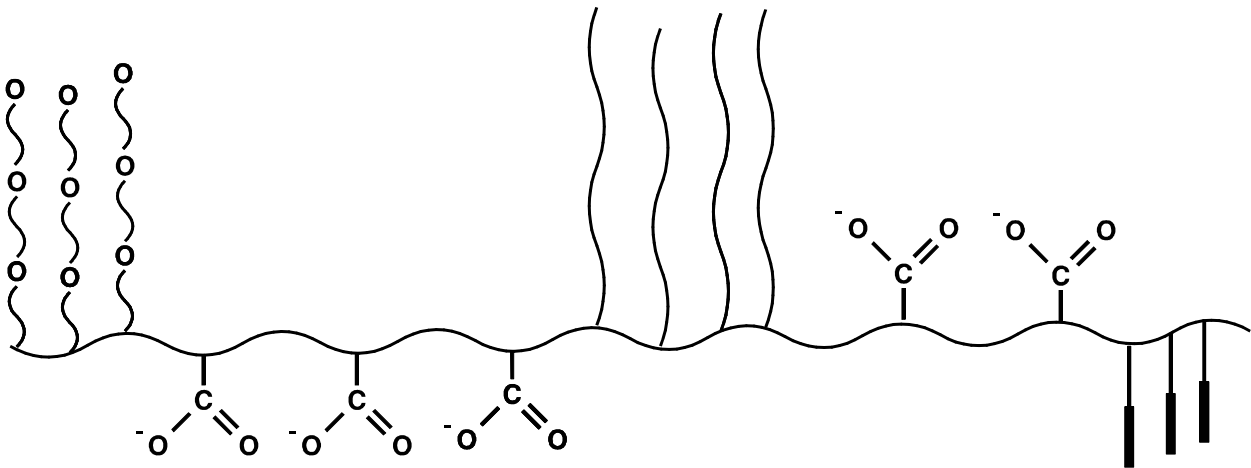
c) *Combination of sterically and electrostatically acting dispersants*

Polymeric dispersing agents unify the properties of classical wetting and dispersing agents. The polyacrylic backbone carries 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 series of polymeric dispersing agents from MÜNZING CHEMIE GmbH:

EDAPLAN® 470 and EDAPLAN® 472

EDAPLAN® 480 and EDAPLAN® 482



	EDAPLAN® 470	EDAPLAN® 472
active substance	approx. 50%	approx. 50%
solvent	butyl diglycol	butyl diglycol
acid number*	110 mg KOH/g	75 mg KOH/g

* The indicated amount of neutralising agent is referred on the products in delivery form.

	EDAPLAN® 480	EDAPLAN® 482
active substance	approx. 85%	approx. 85%
solvent	water	water

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 as unique dispersing and wetting additive in the grinding

The polymeric dispersing agents EDAPLAN 470 and 472 and their solvent free counterparts EDAPLAN 480 and 482 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. This results in an efficient removal of the air from the pigment surface. As a result, higher densities due to lower air contents can be obtained. Especially in lacquers better results of the applied film can be obtained. Additionally, the colour development and the colour strength as well as the gloss can be increased. EDAPLAN polymeric dispersing agents show a very high compatibility with common binder systems and different types of pigment pastes.

7. Use of EDAPLAN as compatibility agent

Apart from the use as unique wetting and dispersing additive the polymeric dispersing agents EDAPLAN can be used in order to improve the compatibility in tinting systems. In this function they lead to a more homogeneous distribution of pigment pastes in basic paints and lacquers. EDAPLAN polymeric dispersing agents therefore increase the compatibility between pigment pastes which are produced with different kinds of dispersing agents and the basic paints and lacquers which contain different kinds of binder systems.

At low dosages of additionally added EDAPLAN (approx. 0.1%) a better compatibility can be obtained, reducing thus efficiently flocculation and flotation processes and other effects which prevent a homogeneous distribution. EDAPLAN compatibility agents do not reduce the scrub resistance and do not tend to foam in comparison to normal wetting agents used in this application.

8. Determination of the dispersant demand

All pigments which are used in paints and coatings systems have various particle sizes and specific surfaces due to their chemical structure and in order to achieve the desired optical and application properties. This means that 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 an indispensable prerequisite to achieve an ideal stabilisation of the dispersed pigments.

In aqueous pigment slurries the required amount of dispersing agent is relatively easy to determine in a first orientative 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:

8.1 Method A

1. Preparation of a pigment slurry:

The pigment content is chosen high enough that a slurry is obtained after dispersion without any dispersing agent which is not fluid any more. Generally, a content of approximately 30% organic pigments and approximately 70% inorganic pigments 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:

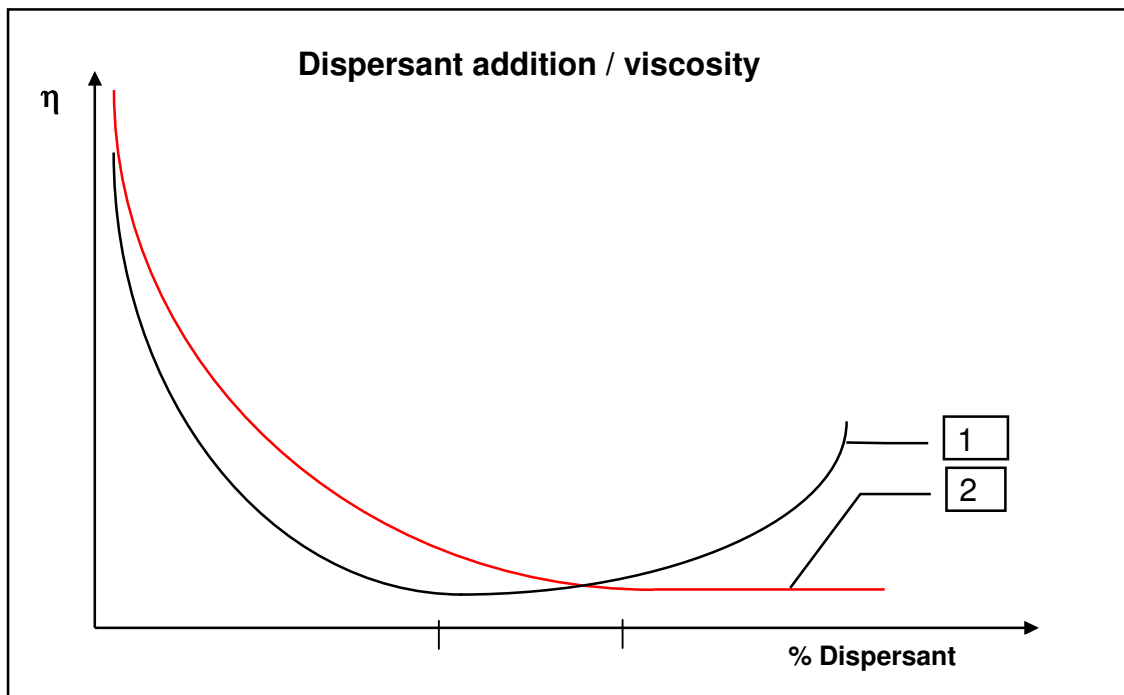
The dispersant is added in portions to the stirred pigment slurry. After the addition and mixing the viscosity is measured at low shear rates (e.g. with a Brookfield viscometer or a cone and plate system).

The dispersant is added until a minimum of viscosity is obtained in the viscosity measurements.

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

Graph [1] is found more or less marked with conventional wetting agents and dispersant 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:

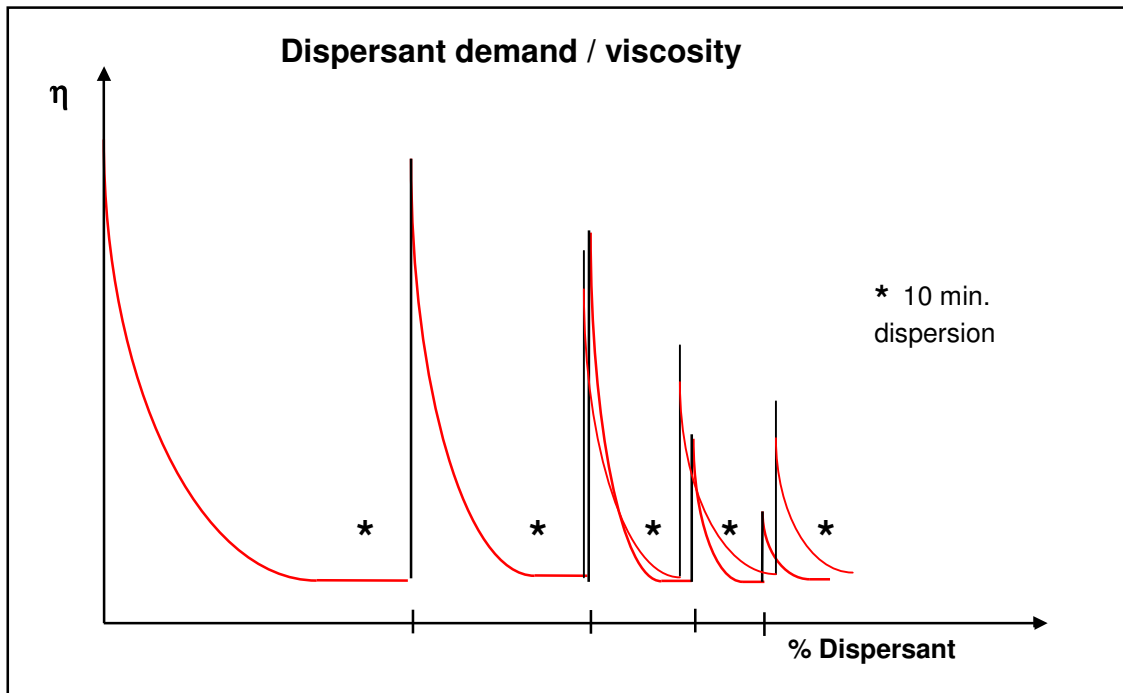
- the optimum degree of grinding is not achieved because the amount of dispersing agent or binder during dispersion is too low (\Rightarrow basics of pigment grinding)
- reagglomeration starts immediately after dispersion is stopped and there is not sufficient dispersing additive or binder to stabilise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

This can be demonstrated if the procedure is changed to the method B described further down in 8.2.

8.2 Method B

1. Dispersion of a pigment slurry with glass balls in a shaking mixer (e.g. „Red-Devil“)
2. Addition of dispersant until a minimum of viscosity is obtained. Dispersion is continued for some more time (approx. 10 min.). In many cases the viscosity again rises so that more dispersing agent has to be added to the slurry. The procedure is continued until a clear minimum in viscosity is obtained.

Generally, for a polymeric dispersing agent the curve of viscosity reduction is displayed like the following graph:



The values of viscosity decrease after each addition of dispersing agent 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.

9. Test method for the determination of the quality of a 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 8.2).

2. Preparation of pigment pastes after the following formulation:

Pigment pastes are prepared with the quantity of dispersing agent which is obtained in the previous experiments. 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 or a sand mill („Red-Devil“ or „Scandex“; in order to obtain comparable values always the same device has to be used). Ratio pigment paste to glass balls = 1 : 1,5 % parts; dispersing time: 40 - 60 min.

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

Ratio pigment to binder (pigment on binder / solids)

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 ⇒ comparison of the degree of grinding
- Colour point, colouring power ⇒ degree of grinding, eventual colour 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. pouring, spraying) ⇒ flocculation, formation of agglomerates
- Transparency or hiding power ⇒ fineness of grinding

10. Comparison table

Many pigments have the same chemical basic structure but treatments of the surface and different coatings from the production can modify the properties of the pigment surfaces significantly. Therefore, different dispersant types and various dispersant quantities can be required for chemically equal types of pigments. The following table shows a comparison between pigments of the same colour index.

Pigment	Type	Dispersant	Dispersant amount [%]*	Gloss [20°]	Haze gloss [20°]
Irgazin DPP red BO	P.R. 254	EDAPLAN® 470	15.0	81.0	98.0
		EDAPLAN® 472	8.0	80.0	115.0
		Competitor	26.0	81.0	82.5
Cinquasia red 750D	P.V. 19	EDAPLAN® 470	(74.0)	66.0	156
		EDAPLAN® 472	15.0	69.0	158
		Competitor	32.0	78.0	102
Cinquasia violett R NRT 201 D	P.V. 19	EDAPLAN® 470	13.0	70.5	215
		EDAPLAN® 472	16.0	79.5	138
		Competitor	61.0	78.5	144
Cinquasia magenta RT 343 D	P.R. 202	EDAPLAN® 470	7.0	82.5	104
		EDAPLAN® 472	8.0	81.0	104
		Competitor	(70.0)	59.5	241
Quindo magenta RV 6843	P.R. 202	EDAPLAN® 470	7.0	50.0	307
		EDAPLAN® 472	7.0	52.0	305
		Competitor	60.0	44.0	324
Kronos 2300	TiO ₂ (Al-Ox.)	EDAPLAN® 470	2.6	83.5	61
		EDAPLAN® 472	3.0	87.0	48
		Competitor	2.3	77.0	111
Kronos 2160	TiO ₂ (Si-Ox.)	EDAPLAN® 470	1.6	91.0	29.5
		EDAPLAN® 472	1.6	88.0	37.4
		Competitor	2.1	89.0	44.1

* in delivery form. The indicates values refer to the quantity which is necessary to obtain the lowest viscosity of the pigment dispersion