PAVE 92

PRACTICAL METHODS FOR DETERMINING THE TINTING STRENGTH OF PIGMENTS IN CONCRETE

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Summary

The tinting strength of the pigments used to colour concrete products such as pavers and roof tiles is of particular significance. Pigments with high tinting strength will create a more intense colour than those with lower tinting strength, even if the same quantities are used. This means that smaller amounts of the pigments with high tinting strength are needed to create a certain colour intensity. Colorimetry makes it possible to measure tinting strength under practical conditions in the laboratory. To ensure the accuracy of the colorimetric measurements, the concrete surfaces should be as homogeneous as possible and completely free from efflorescence. The method described in this paper makes this possible and is an interesting complement to standard methods used to measure tinting strength in building materials.

The tinting strength values for two pigments of similar shade can be used to calculate the pigment concentrations required to create the same colour intensity.

It is also possible to use tinting strength measurements to draw conclusions about the dispersibility of a pigment in concrete under certain variable mixing conditions. It is interesting to test the pigments in the form of powders, aqueous suspensions or granules.

1 Introduction

The German standard DIN 55986 defines tinting strength as a measure of the ability of a colorant, on the basis of its absorption capacity, to colour other substances. At first glance this definition might appear to be somewhat complicated so what does it actually mean?

The manufacture of a red concrete paver, for example, requires the addition of a colorant, usually referred to as a pigment. Red iron oxide pigments are generally used. These differ in particle size and, to a certain extent, purity. Used in concrete they will result in various shades of red, for example, a yellowish or bluish red. They also have different tinting strengths. The higher the tinting strength of a pigment is, the less of that pigment will be needed to create a certain colour intensity. Tinting strength is therefore a particularly important factor in the cost-effective use of pigments in concrete and other substances such as plastics and coatings. Generally speaking, tinting

Standards dealing with pigments therefore include methods for the measurement of tinting strength. In most cases the shade is measured at the same time. Information about the tinting strength of a pigment must always include the medium in which the measurement was taken. Paint systems such as alkyd resins are normally used. The simple reason for this is that the test pigment is easily dispersed in resins to produce smooth, clean paint films suitable for measurement with a colour measuring device.

Anyone who works with pigments knows from experience that the results of tinting strength and shade tests carried out in paints cannot simply be applied to other media such as concrete. As far as shade is concerned, a paint binder is generally colourless, i.e. it has no natural colour of its own. In concrete, where the cement used as the binder is pigmented, the shade varies depending on the natural colour of the cement itself, whether it is a white cement or a dark portland cement. In addition, concrete contains aggregates as large as 10 mm or more so it is small wonder that the results measured in paints cannot be applied to concrete.

This fact is recognized in the various national standards governing the use of pigments in concrete.

The German standard DIN 53237 "Pigments for cement and lime-bound building materials" describes a method for measuring tinting strength. The test pigment is mixed dry with white cement or barytes and the tinting strength determined visually or colorimetrically. This method is quick and easy and the values obtained approximate those found in concrete.

The British standard BS 1014: 1975 describes a similar method. The pigment and the cement are either mixed dry between two glass tiles or with water and then assessed visually.

In both methods the pigment and cement or barytes are mixed thoroughly with the aid of glass beads. The best results are achieved using a shaking machine. However, in both cases no aggregate is added and it is this which ensures the optimum dispersion of pigments in concrete.

It is therefore necessary to develop a method where the pigment is mixed with cement, various aggregates and water, and the tinting strength is measured after the concrete has hardened, preferably not visually but with a colour measuring device.

Just such a method is described in the following paragraphs. It is intended to complement the existing methods as laid down in various standards.

2 Method for determining the relative tinting strength

2.1 Basic principles

Tinting strength is always determined relatively, i.e. the tinting strength of a reference pigment is taken to be 100 % and that of the test pigment as higher or lower in relation to this.

When measuring the relative tinting strength of the pigments commonly used in concrete in accordance with German standard DIN 55986, the tristimulus value Y = Ry is used as the tinting strength criterion. Ry characterizes the lightness of a colour. Numerous comparative studies have shown this value to be the most closely compatible with the visual assessment made by colorists.

As most modern colour measuring devices measure Rx and Rz as well as Ry, other colorimetric values are determined in addition to the relative tinting strength (a*, b*, L*, C*) which aid a more complete characterization of a colour. They are calculated from Rx, Ry and Rz.

It is not possible within the scope of this paper to go into great detail about these colorimetric values. I can refer you to manufacturers' information [1], papers [2] and general trade publications [3].

A basic knowledge of the principles of colorimetry are sufficient for it to be of some use in solving the problems encountered with coloured concrete. As the human eye is very sensitive, it is to be recommended that any values given are always confirmed and compared by visual assessment and not merely taken at face value.

2.2 Manufacture and measurement of concrete samples

The surfaces to be measured by colorimetric methods must be free from any efflorescence (calcium carbonate) which could produce wildly inaccurate results. It is not recommended that the concrete samples are manufactured and then broken, the measurements being taken on the broken surfaces, because most colour measuring devices only function properly on a smooth surface where no light can seep in at the sides. Only those instruments which operate without touching the sample, and these are few, do not have this disadvantage.

Careful formulation and selection of the various aggregates (see appendix) will result in relatively smooth concrete surfaces free from efflorescence. The next paragraphs describe only the principles of the method. The exact procedure can be found in the appendix.

It is also essential that the aggregate used is no larger than 2 mm. A certain quantity of extender must also be to 2 mm quartz powder to an equivalent calcium silicate powder. Experiments showed that this had no effect on the results. Any aggregate used should have little natural colour, i.e. it should be as close to white as possible. It is therefore advisable to use standard, or at least washed, sand.

White cement should be used as only then will the reflectance values (Ry greater than 10 if possible) be such that the colour measuring device can operate with sufficient accuracy.

Hardening is especially important for reproducibility. It is obvious that temperature, relative atmospheric humidity and time will have an effect. However, it has also been found that the amount of available CO_2 has an influence on these sensitive measurements. Many laboratories are not equipped with a climatic chamber and, even if they do have one, the CO_2 content cannot be kept uniform because of the varying number of concrete samples and the constant opening and closing of the door. For this reason, we employ a simple but very practical method.

Four concrete samples are placed on a wooden rack (Fig. 1). This is then place in a polythene bag which is sealed (Fig. 2). There are sidepieces on the rack to keep the polythene bag from touching the concrete samples. This prevents condensation water from soaking into the concrete. The bag is then placed in a drying cabinet heated to 30 °C so that the concrete hardens. This very simple method allows the maintenance of constant CO_2 content, relative humidity, temperature and hardening time.

Care should be taken that the concrete samples are stood on their sides to harden. The sides are unsuitable for colorimetric measurement as they undergo slight abrasion when they are removed from the mould with the result that they are darker. Each sample is measured at six points, three on the top and three on the bottom. Four samples are produced from each mixture, giving a total of 24 measurements. A properly programmed modern colour measuring device will carry out these measurements relatively quickly. Unfortunately, such a large number of measurements are required when testing concrete as, even with the greatest care, surface defects are unavoidable. The only way to compensate for this is to take multiple measurements.

3 Do laboratory results correlate with practical conditions?

3.1 Comparison of a 2.5 kg mixture with a 60 kg mixture

Tests were carried out to see whether the results obtained in a laboratory with a 2.5 kg mixture and a small mixer correlate with those obtained in a pilot plant using a 60 kg mixture and an intensive mixer. The same formulation was used in both cases.

Five different iron oxide black pigments were used, four in powdered form (P1 to P4) and one as a 50 % aqueous suspension (S1). Table 1 lists the values recorded. Two 60 kg mixtures were produced in the intensive mixer. A good correlation is found between the average values from these two mixtures and the values obtained under laboratory conditions.

Table 1

Relative tinting strength (%)
Comparison of laboratory and pilot plant mixtures
Mixing time: 120 s

No.	Method	Measuring pts.	P1	P2	P3	P4	S1
1	Lab. (2.5 kg)	96	100	77	72	48	58
2	Pilot (60 kg)	30	100	76	70	50	57
3	Pilot (60 kg)	30	100	77	75	52	59
4	Mean 2 + 3	60	100	77	73	51	58

P1 to P4 = different iron oxide black pigments
S1 = aqueous iron oxide black suspension
P1 = reference = 100 %

3.2 Using the laboratory method to test extruded concrete roof tiles

Tests were carried out to determine whether the results of laboratory tests can be applied to the manufacture of concrete roof tiles. A cement/aggregate ratio of 1 : 3 was used with a water/cement factor of 0.39 and a pigment concentration of 2 %. A low pigment concentration was selected so that good measurable reflectance was obtained despite the use of black pigments. Four powder pigments were used (P1 to P4). Two different cements were chosen - a grey portland cement and a white cement. Two mixtures were produced with each, sometimes both on the same day and sometimes on two separate days. These were then used to make roof tiles.

Table 2

Relative tinting strength (%)
Comparison of concrete roof tiles manufactured in the laboratory and pilot plant

No.	Method	Mixture	Measuring points	P1	P2	P3	P4
1	Lab.	4	96	100	77	72	48
2	Roof tiles grey, 2 days	2	40	100	78	76	55
3	Roof tiles white, 1 day	2	40	100	74	68	44
4	Roof tiles	2	40	100	80	76	53
5	Mean 2 to 4	6	120	100	77	73	51

P1 to P4 = different iron oxide black pigments
P1 = reference = 100 %

The results are summarized in Table 2. The roof tiles were manufactured using a pilot plant extruder. They were hardened under constant conditions in a steam cabinet. Unfortunately, it is not possible to keep all the parameters which affect colouring as constant under pilot plant conditions as under laboratory conditions. For this reason there is a wide discrepancy between the individual values measured on the roof tiles. However, the mean values (line 5) show good correlation with the mean values obtained in laboratory tests (line 1).

4 Examples of application

4.1 Comparison of two iron oxide red pigments of differing tinting strength in concrete pavers

The following situation might arise in a plant manufacturing concrete pavers. Red pavers are produced using 5 % of a pigment with a relatively low tinting strength (R2). A second series of pavers with a facing is made using 7 % of the same pigment. The question is, what must the pigment concentration be in both cases to produce the same colour using a pigment with a higher tinting strength? It must be added that the two pigments R1 and R2 are very similar in shade, a prerequisite in such comparisons, but R2 has a bluer undertone than R1.

Using the laboratory method already described, a relative tinting strength of 64 % is determined for R2 with R1 taken as the reference pigment at 100 %. It can be calculated that, instead of the 5 % and 7 % concentrations needed for R2, only 3.2 % and 4.5 % of R1 are required. Table 3 lists the colorimetric values.

Table 3

Colorimetric values for iron oxide red pigments R1 and R2 after adjustment because of their differing tinting strengths

	L	a	b	ΔL	Δa	Δb	ΔE	Rel. tinting strength
7.0 % R2	42.3	7.2	4.2					
4.5 % R1	42.1	8.1	8.1	-0.1	0.9	3.8	4.0	100.6
5.0 % R2	42.6	6.7	3.3					
3.2 % R1	43.5	8.6	5.6	0.9	1.8	2.3	3.1	92.0

(Laboratory method: rel. tinting strength of R2 = 100 % and R1 = 64 %)

After adjusting the pigment concentrations because of the differing tinting strengths of the two pigments, a tinting strength of 100 % should theoretically have been measured for both pigments. However, the actual values of 100.6 % and 92.0 % are very satisfactory. The overall colour differences ΔE of 4.0 and 3.1 are relatively high but are due to the fact that the shades of the two pigments are not exactly identical. This is the limitation in using this method to match or adjust pigment concentrations. It is therefore necessary that the pigments used are as similar in shade as possible.

Fig. 3 shows the red pavers with 7.0 % R2 and 4.5 % R1 in the facing and those with 5 % R2 and 3.2 % R1 in the concrete mixture.

4.2 Dispersion of pigments in concrete

Determination of the relative tinting strength by the method described can also be used for other interesting applications. As a general rule, the relative tinting strength increases with dispersion time until the maximum is reached. This means that the relative tinting strength is an excellent test parameter for the degree of dispersion.

In the examples described in the following paragraphs, poor mixing and dispersion conditions were purposely chosen. The aggregate, pigment, cement and water were mixed at once. Dispersion tests were carried out after total mixing times iron oxide red, was used in both powdered and microgranulated (particle size 100 - 200 µm) forms. The diagram in Fig. 4 shows that, under these conditions, the microgranulated pigment disperses more rapidly in the concrete than the powder pigment.

5 Appendix

Manufacture of concrete samples for determining relative tinting strength

Test mixture

500 g white cement (portland cement 45)
200 g calcium silicate powder
1,200 g quartz sand, 0.2 - 1 mm
600 g quartz sand, 1 - 2 mm
175 g water (water/cement factor = 0.35)
6 g pigment (1.2 %, calculated on cement content)

Fig. 5 shows the Toni mixer used. This equipment is in widespread use in the concrete industry (in accordance with DIN 1164, BS 4550 Part 3 and ASTM C 305-80).

The quartz sand and calcium powder were taken and the pigment added as either powder, granules or aqueous slurry. If powder or granules are used, the water is then added. If a slurry is used, the necessary amount of water needed to make up the total requirement is added. The components are mixed slowly for 30 s. The cement is then added and slow mixing is continued for a further 30 s. The mixing speed is then doubled for 60 s. The total mixing time is 120 s.

300 g of this mixture is pressed in a steel mould at 32.5 N/mm^2 to form a sample 5 x 10 x 2.5 cm (Fig. 5). Four samples are usually produced from each mixture. These are placed on a wooden rack (Fig. 1) and sealed in a polythene bag (Fig. 2). The whole process from making the samples to sealing them in the bag should be carried out as quickly as possible.

The samples are then placed in a drying chamber (30 °C) where they harden in 24 h ± 2 h. The polythene bag is then removed and the samples left to stand for 2 h in the laboratory. They are then cured for at least 10 h, 20 h at the most, in a drying chamber at 50 °C. Before measurements are taken with a colour measuring device (d/8° geometry, standard illuminant C with inclusion of gloss, aperture 5 mm) (Fig. 7), the samples must be cooled to room temperature.

Experience has shown that this work is best and most quickly carried out be three people.

Determining the relative tinting strength is done in accordance with DIN 53234 "Determining the relative tinting strength in white reductions using the photometric method". Calculation of the CIELAB values is carried out in accordance with DIN 6174 and DIN 53140. The test procedure is shown in Table 4.

Given the care required in producing the samples and the number of measurements which must be taken, this is not a quick method. An experienced tester will require approximately 2 h to test one pigment (4 samples from each of 2 mixtures). Because of the long hardening and curing times, it takes at least two to three days to obtain the test results. The method is a good complement to existing standard test methods as far as fundamental questions are concerned.

To determine the accuracy of the method, two test series of ten mixtures each were run. The standard deviation was 0.19 and 0.27 respectively. The mean Ry for series 1 was 15.59 ± 0.19 and for series 2 15.56 ± 0.27. If the reflectance values Ry are approximately 15, the difference is therefore around ± 1.5 %. At the very worst, the maximum difference ΔRy is about 3 %. Before the mean values could be calculated for mixtures 1 and 2 as shown in Table 4, it was decided that ΔRy had to be less than or equal to 3 %. Otherwise a new mixture would have to be prepared.

The standard deviation of the relative strength is, however, lower. The results of the two test series described result in a standard deviation of ± 3 units.

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Table 4 Test procedure

Bezugspigment = Reference pigment
Probepigment = Test pigment
Mischung = mixture
Steine = pavers
Messung = measurement
Messpunkte = measuring points
Mittelwert = mean
ja = yes
nein = no
neu präparieren = prepare new mixture
relative Farbstärke über Ry =
relative tinting strength over Ry
Cielab-Werte = CIELAB values
Farbdifferenzen = colour differences

Fig. 1: Wooden rack with four samples

Fig. 2: Wooden rack sealed in a polythene bag

Fig. 3: Red concrete pavers

Alle Dezimalkommas durch Dezimalpunkte ersetzen!
Pigment concentration

Iron oxide red pigments

Fig. 4: Dispersion curve for an iron oxide pigment (powder and microgranules) in concrete

Tinting strength after 150 s

granules
powder

Toni mixer

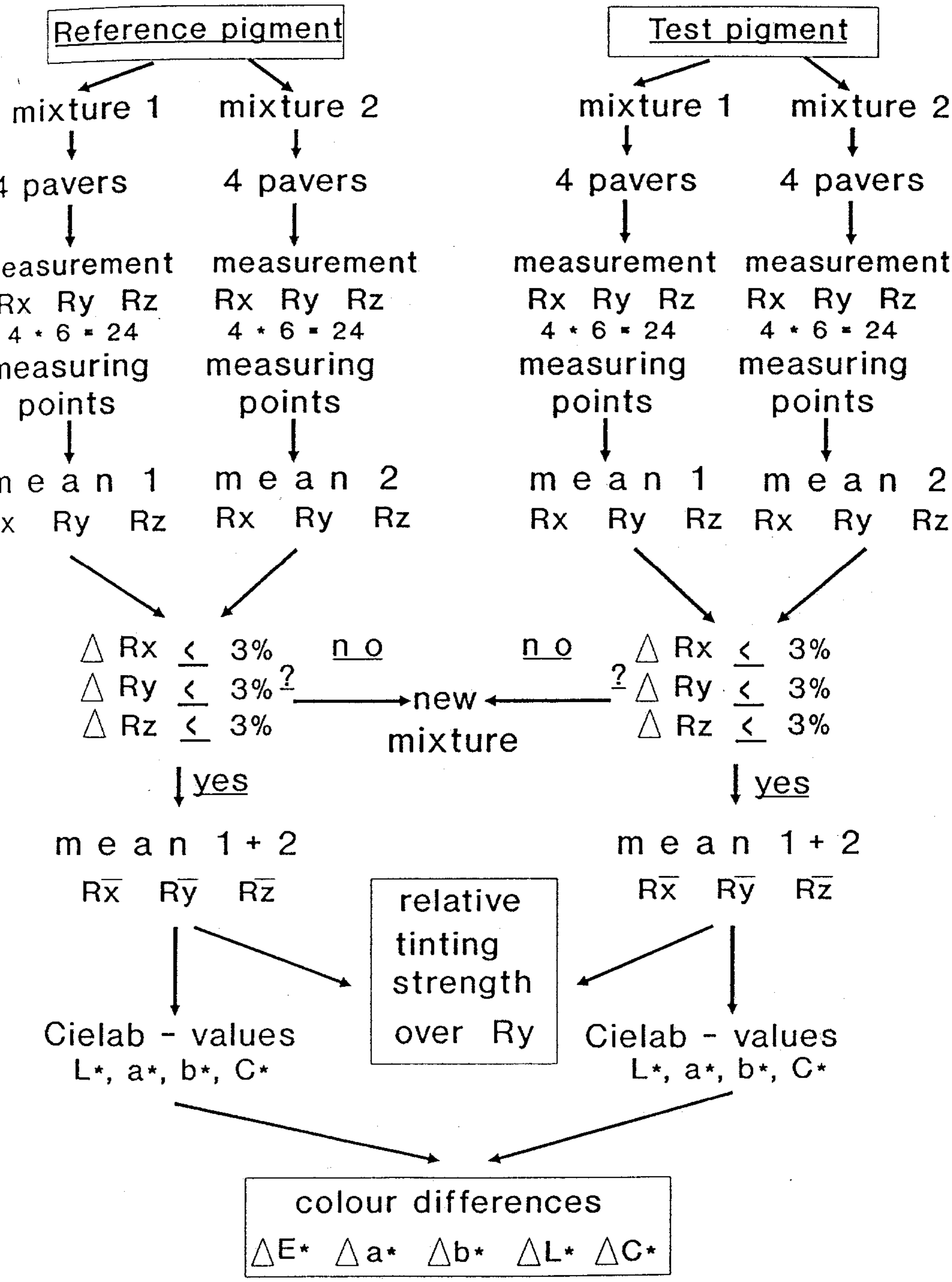
Total mixing time (s)

Fig. 5: Toni mixer

Fig. 6: Press for producing concrete samples

Fig. 7: Colour measuring device

ble 4 **Test procedure**



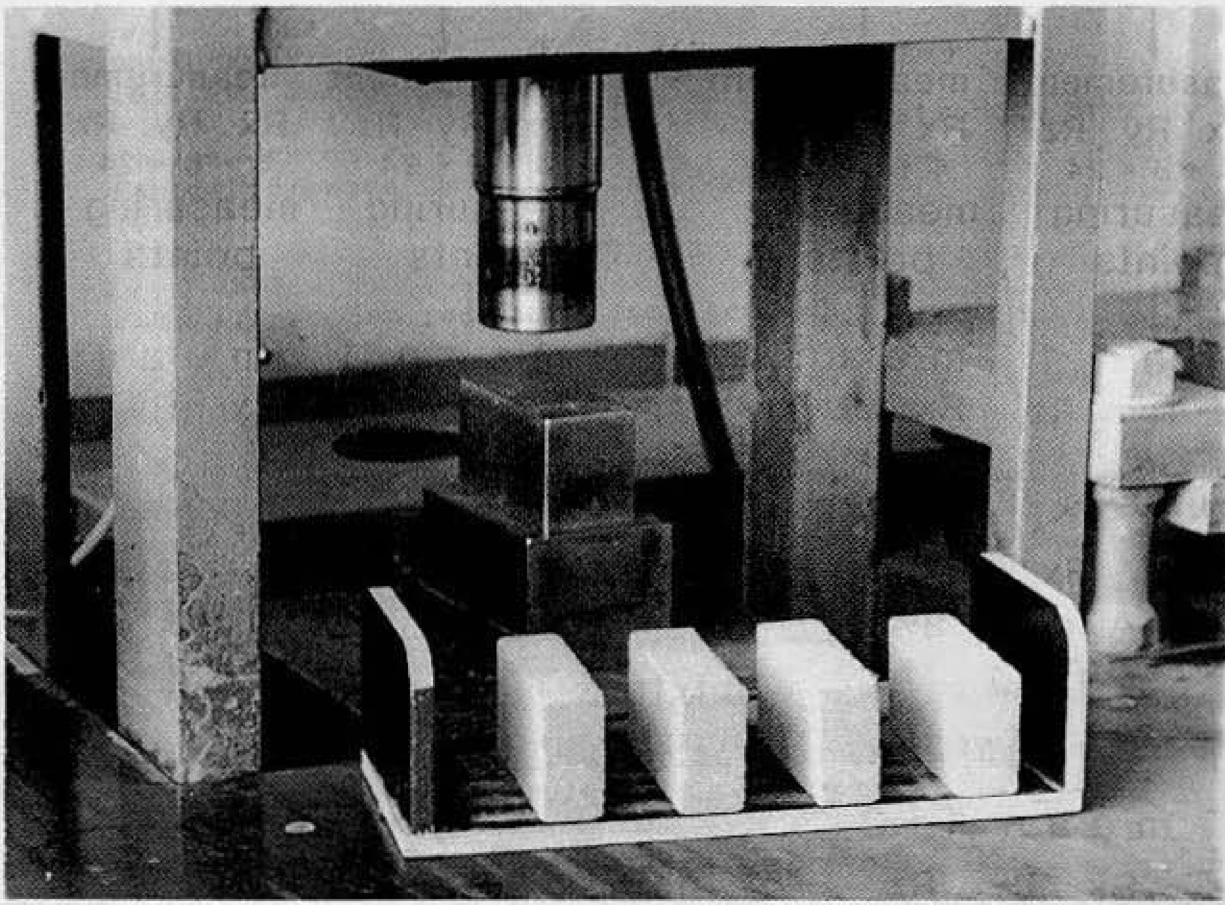


Fig. 1: Wooden rack with four samples

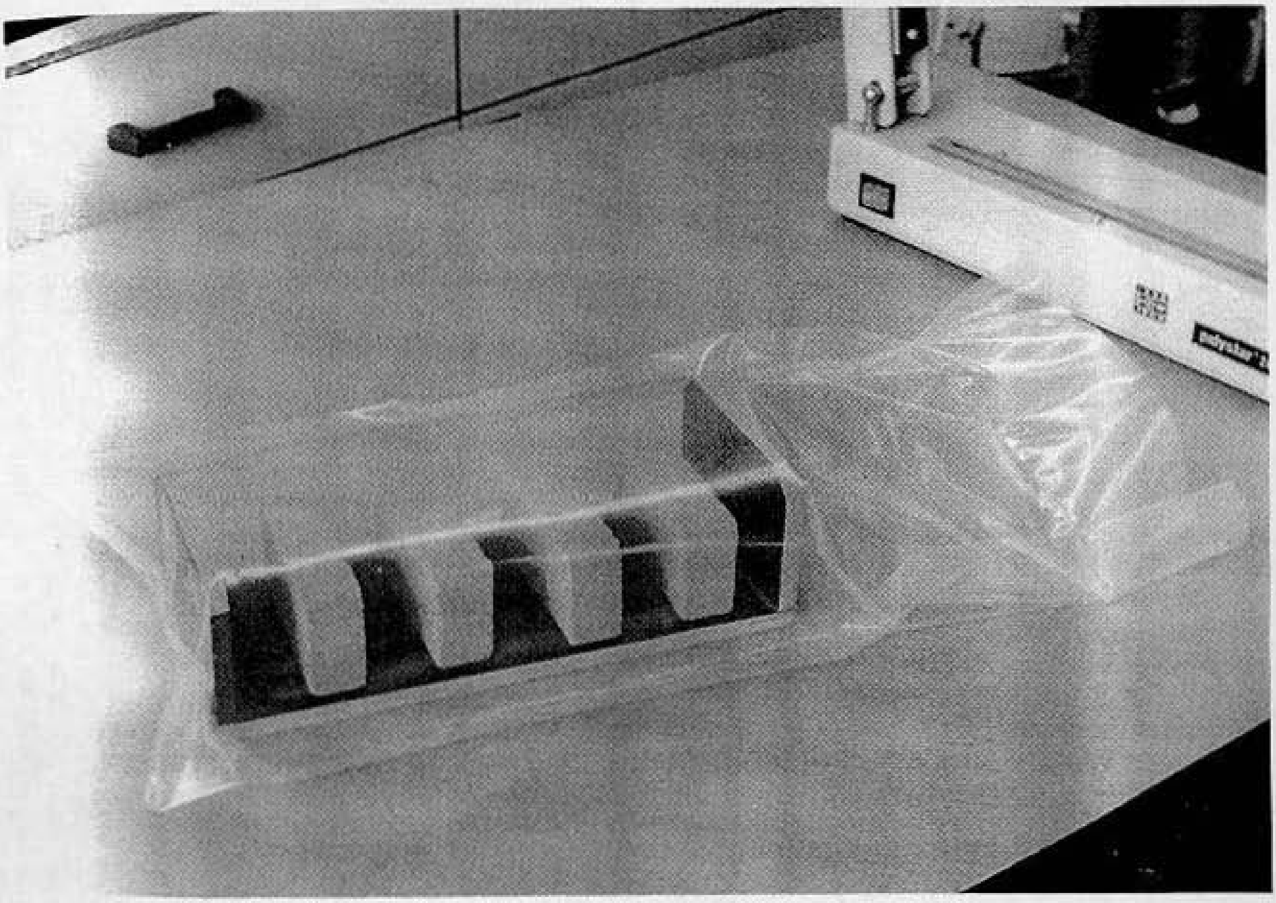
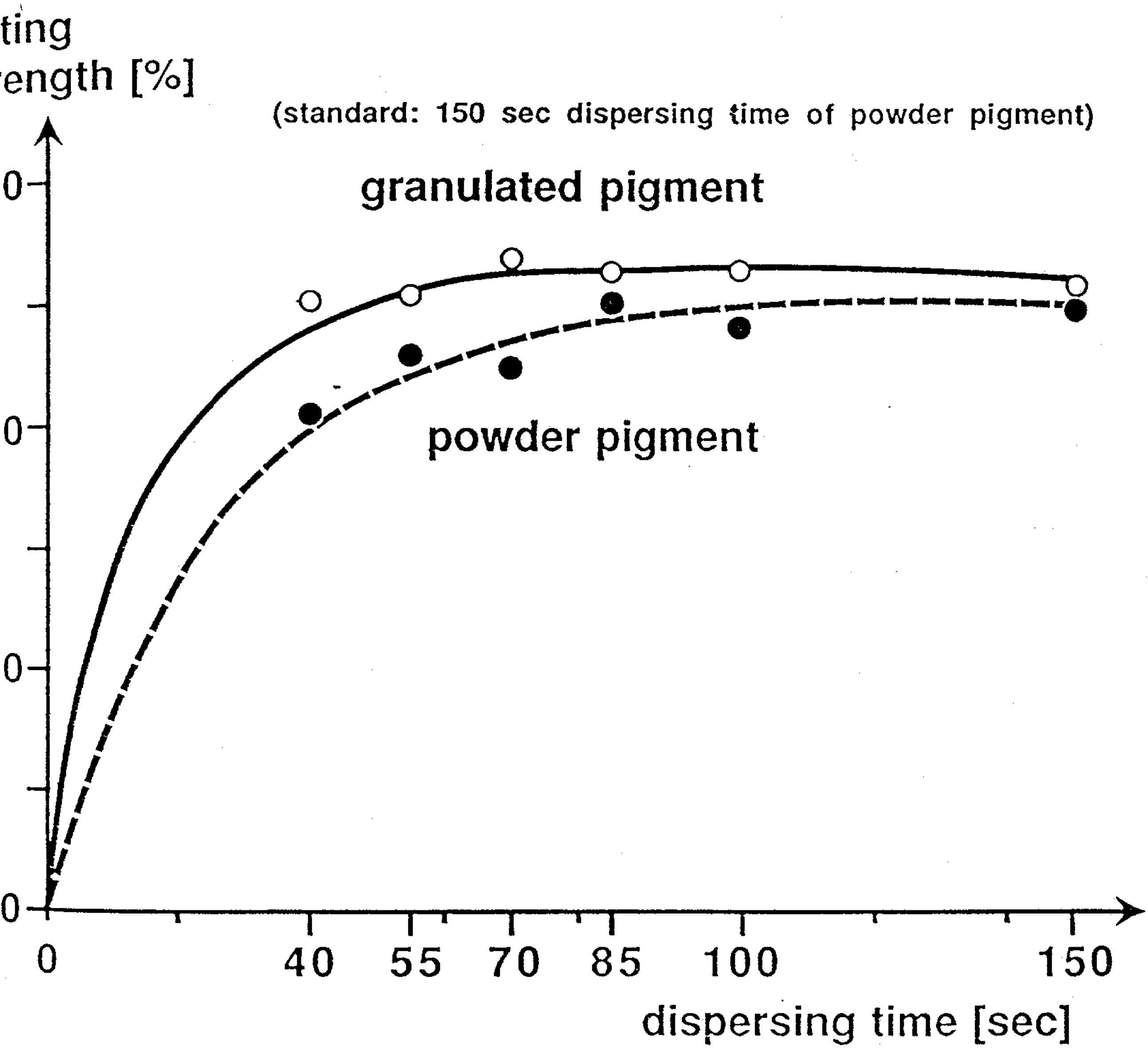


Fig. 2: Wooden rack sealed in a polyethene bag

Pigmentierungshöhe = Pigment concentration (calculated on cement)

Eisenoxidrotpigmente = Iron oxide red pigments

Fig. 3: Red concrete pavers



Dispersibility of granulated iron oxide red in comparison to powder

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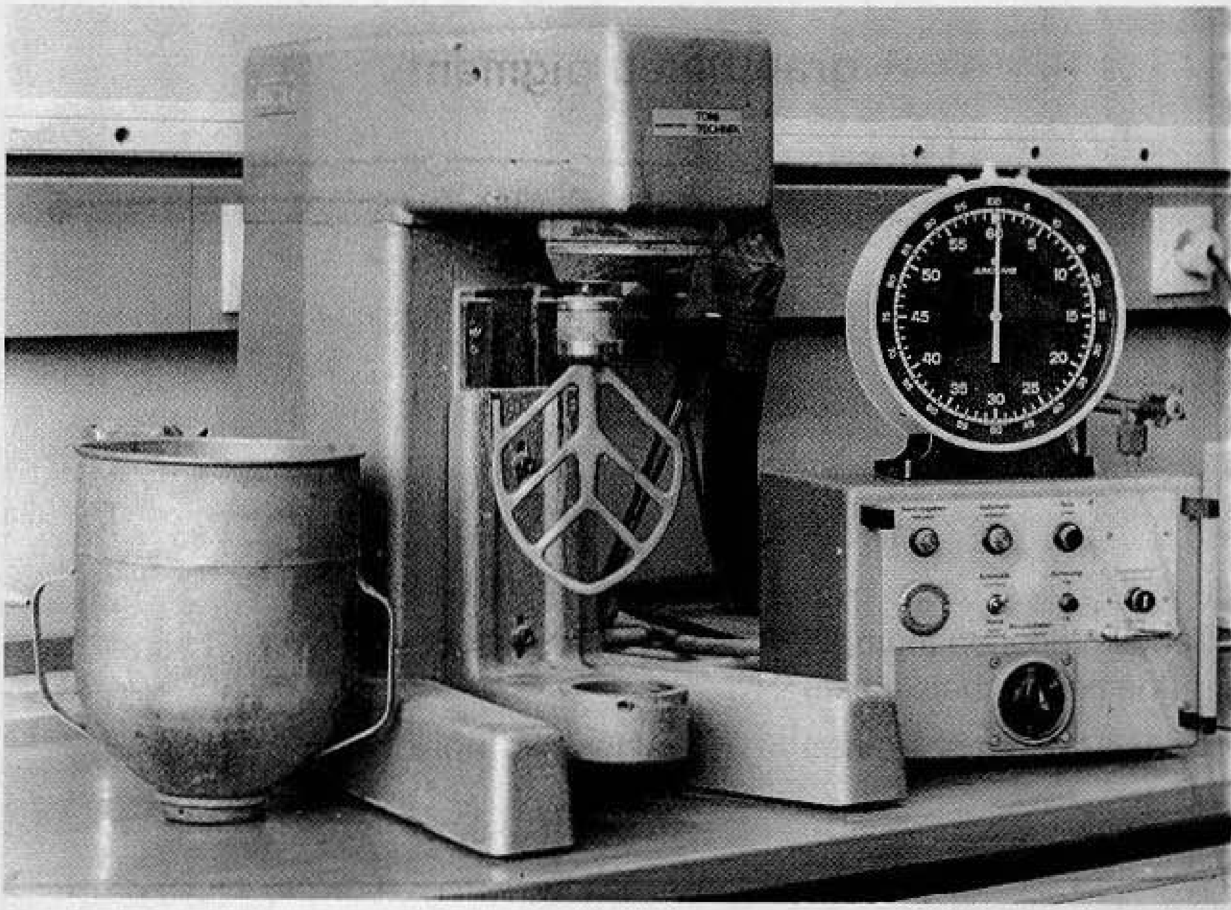

Fig. 5: Toni mixer

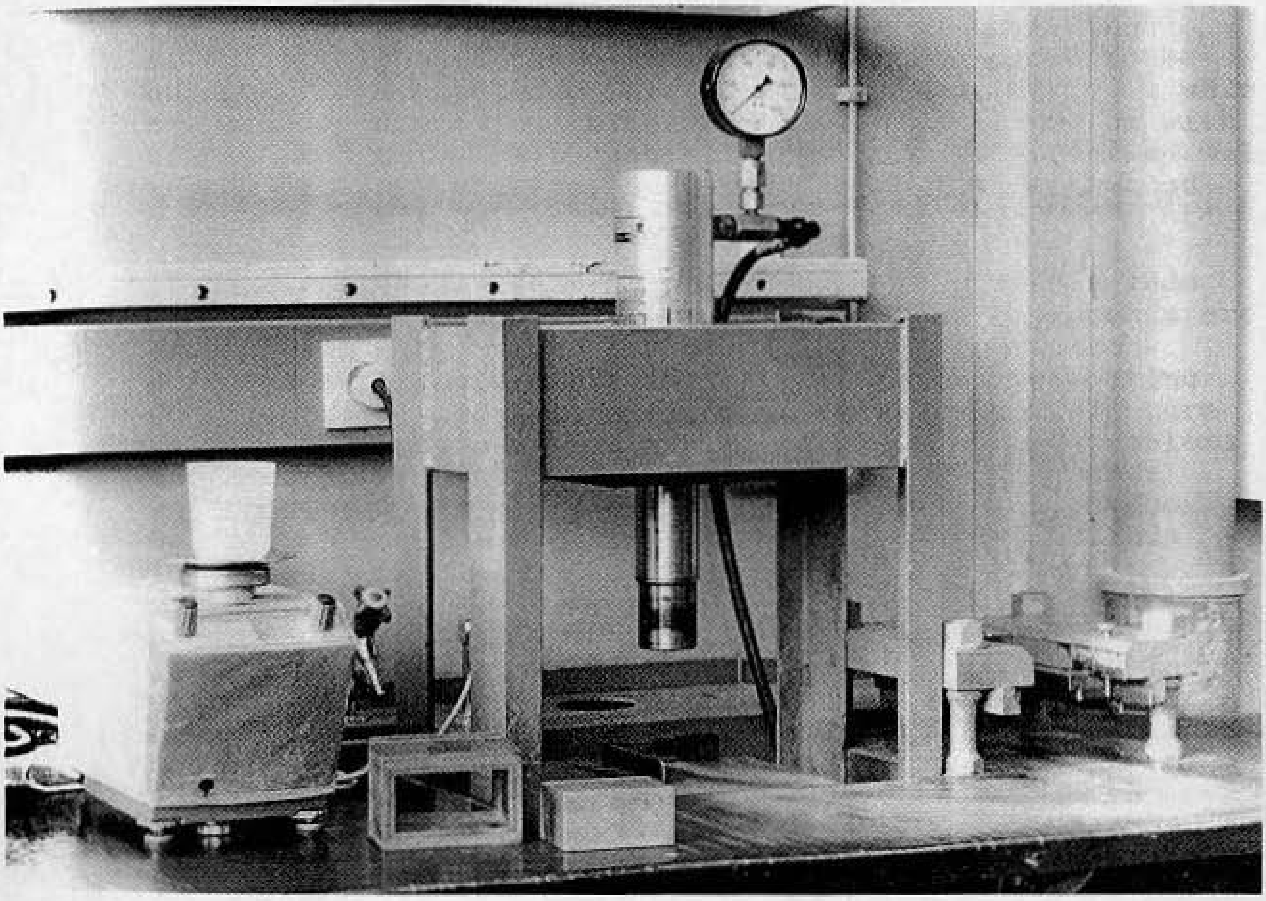

Fig. 6: Press for producing concrete sample

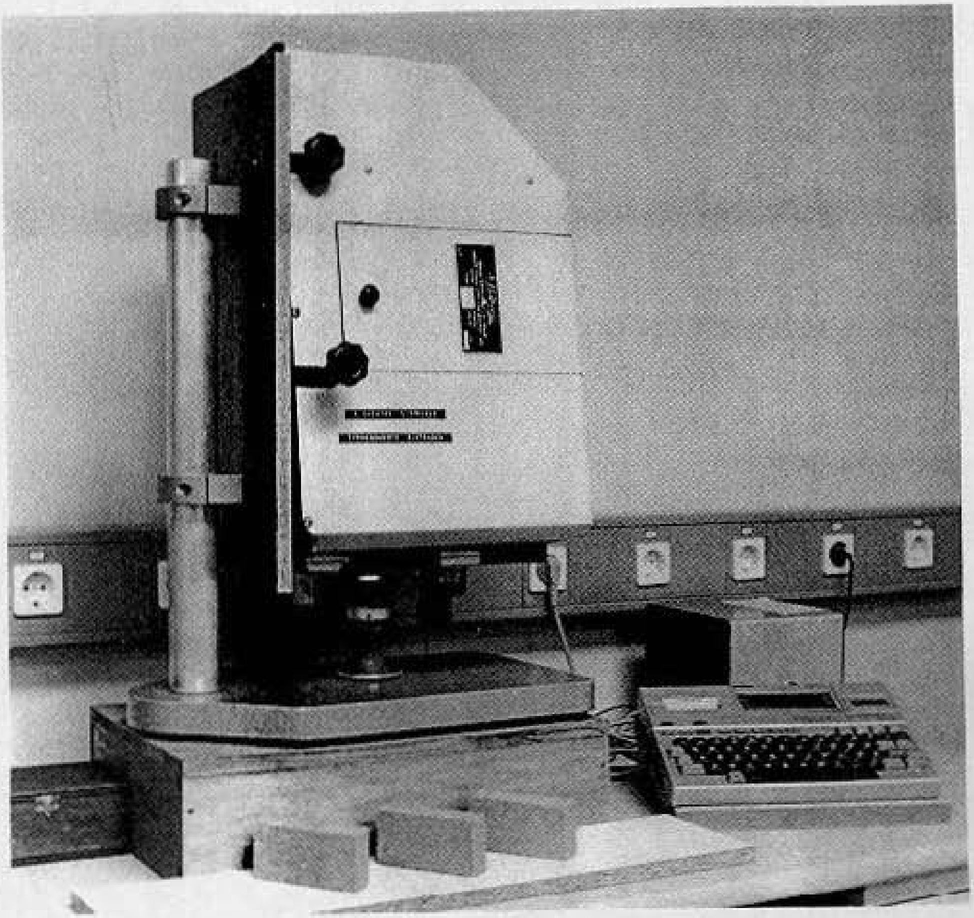

Fig. 7: Colour measuring device