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# IMPROVEMENTS TO THE METHYLENE BLUE DYE TEST FOR HARMFUL CLAY IN AGGREGATES FOR CONCRETE AND MORTAR

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**ABSTRACT**

Clay minerals found in natural aggregates are associated with harmful effects in concrete and mortar. The methylene blue dye adsorption test is included in the draft European Standards for the determination of harmful fines in aggregates for concrete and mortar. This test identifies deleterious clay minerals but does not give results in proportion to the damaging effects that different clay minerals have on the strength of concrete and mortar. This paper outlines some preliminary research which identifies a combination of clay treatment and a dye that can be used to test clays and rank them in order of their harmfulness. Further work is required to include a broader range of clay types and to develop the test for use on aggregates. © *1998 Elsevier Science Ltd*

## Introduction

Clay minerals are commonly found in the fine fraction (sub-63 μm material) of natural aggregates. In general, the presence of excessive quantities of clay is considered harmful in concrete and mortar. The extremely fine particulate nature of clay minerals and the activity of their surfaces generally increase the mix-water required to provide a concrete or mortar of given workability. This has implications for the strength, durability and volume stability of the hardened concrete or mortar.

Briefly, clay minerals are crystalline hydrated alumino-silicates. The smectites comprise repeat units of silica, gibbsite (aluminium hydroxyl groups) and silica with adjacent silica layers joined by Van der Waals bonds. Substitution of aluminium for magnesium in the gibbsite sheet and aluminium for silicon in the silica sheet results in a net negative charge in the smectites, imparting a high cation exchange capacity. In addition to a high cation exchange capacity, the smectites can increase the inter-layer spacing between silica sheets, swelling as they adsorb water. Kaolinites comprise repeat units of silica and gibbsite; they lack the inter-layer spacing found between adjacent silica sheets in smectites and their cation exchange capacity is less than that of the smectites and is predominantly due to causes other than substitution.

Because of their ability to exchange cations, clay minerals adsorb methylene blue (a

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cationic dye) from aqueous solution. This ability of clays to adsorb dye onto active surfaces is the basis of test methods for measuring cation exchange capacity and surface area (1,2) and for detecting the presence of clay minerals.

The test method commonly used for detecting clay fines in aggregates is based on a titration, adding a known quantity of dye to a sample (normally an aqueous suspension of fines and sand, although water extracted fines have been tested) until an end-point is reached where no more dye can be adsorbed. The end-point is determined, using a spot-test, by taking drops of water, together with dye stained, suspended fines, from the test portion and placing them on a filter paper. As long as the fines are capable of adsorbing dye, the spot-test reveals a circle of blue-stained fines surrounded by a colourless ring of water absorbed into the filter paper. The end-point is reached when the fines can adsorb no more dye, the excess being drawn away from the spot of fines to form a blue ring or "halo."

### Some Problems Associated with Detecting Clay Using the Methylene Blue Dye Test

When combined with a measure of fines content, the methylene blue dye test provides a useful indication of the overall clay content of an aggregate. However, there are problems with interpreting the results, namely that the adsorption of dye by different clay minerals is not in proportion to the damaging effect that those clay minerals have on concrete and mortar (3). Additionally, the methylene blue dye test alone cannot distinguish between the three types of clay mineral prevalent in the fines of natural aggregates. Table 1 is based on rules of thumb suggested by Pike (4) for strength losses in cement composites contaminated with the three clay minerals commonly associated with natural aggregates.

At present, results of methylene blue dye tests on illite and smectite are not in direct proportion to the damaging effect that these clays have when present as contaminants in aggregate for concrete and mortar. The adsorption characteristics of illite and smectite need to be reduced in order to bring them into line with their effects on strength reduction. There is scope for improving the application of the dye test and the meaningfulness of the results that it produces. The research described in this paper aims to achieve significant improvements in this area.

## Experimental Work

### Mortar Mixes

In the work reported here, a limited series of mortar mixes dosed with clay was made and tested to confirm the work of other authors on the strength reductions associated with clays in aggregate. Some novel experiments using methylene blue dye in mortar were undertaken in an attempt to explain the behaviour of clays in mortar.

1:4 cement:sand mortars, proportioned by mass, were made to both constant water:cement ratio and constant workability. The sand was dosed with varying quantities of oven-dried kaolinite and smectite, which were mixed into the dried sand before the addition of cement and water. Non-clay fine material (used as an "inert" replacement for a fraction of the sand in mixes without added clay) was manufactured by grinding the flint concreting sand to passing 63 $\mu$m (the finest mesh, standard sieve size available) using a ball mill.

Cubes (100 mm) were cast for each mix, de-moulded at 24 h, cured under water and tested

TABLE 1
Methylene blue values of clays and reductions in strength of concretes and mortars.

| Clay mineral | MB value g(dye)/kg(clay) | Strength reduction* (percent for addition of 1% of clay by mass of cement) |
|---|---|---|
| Kaolinite (K) | 13 | 1 |
| Illite (I) | 65 | 2 |
| Smectite (S) | 260 | 4 |
| Ratio K:I:S | 1:5:20 | 1:2:4 |

*Strength reductions for concretes and mortars made to constant workability.

for compressive strength at 28 days. One set of mortar mixes, to constant water:cement ratio, was made using mix-water containing sufficient methylene blue dye to “neutralise” the clays present in the sand.

While preparing mortars with different clay types and contents, it became apparent that, for mortars made to the same consistency (measured by the plunger penetration method (7)), workability was very different. Initially, workability was not measured, but was controlled using the subjective opinion of the technician. These mixes cannot be compared with later mixes, where workability was controlled using the flow table method. For these mixes, a measure of workability was made using a concrete flow table (the only equipment immediately to hand).

The concrete flow table was adapted for use on the mortars by using a small, open-ended, slightly tapering plastic cone 11.8 cm in height and 8.8 cm and 9.2 cm internal diameter. The mortar was compacted into the cone in three layers, and each layer was tamped with 12 blows from a steel tamping rod, the final layer being struck level using the tamping rod. The plastic cone was drawn off smoothly over a period of 5 s; the table was then raised and dropped 5 cm five times and the diameter of the mortar pat was measured in two directions. Air content of the mortar was measured using the density method (8).

TABLE 2
Dyes.

| Dye | Molecular weight | Form | Dye purity | Quantities used | Dye concentration |
|---|---|---|---|---|---|
| Methylene blue | 340 | Powder | 85% | 11.5 g(dye)/L | 9.8 g(dye)/L |
| Cartasol red | 850 | Liquid | 9% | 114.1 g(liquid)/L* | *ca* 10.3 g/L |
| Cartasol turquoise | 1150 | Liquid | 10–15% | 118.1 g(liquid)/L* | *ca* 14.8 g/L |
| Cartasol blue | 1500 | Liquid | 10–15% | 119.3 g(liquid)/L* | *ca* 14.9 g/L |

*The cartasol dyes were supplied in liquid form with imprecise information on dye concentration. The solutions for the tests were made up to volume with water in proportions according to the original work done by Pike (4).

TABLE 3
Properties of mortar made to constant workability.

| Fines added, % by mass of cement | | Water-cement ratio | Plunger mm | Flow mm spread | Air content | 28 day $N/mm^2$ | Strength % loss |
|---|---|---|---|---|---|---|---|
| None | 0 | 0.761 | 26 | / | 2.3 | 33.2 | 0.0 |
| Kaolinite | 4 | 0.794 | 38 | / | 1.7 | 29.6 | 10.8 |
| Smectite | 4 | 0.901 | 36 | / | 1.9 | 20.2 | 39.2 |
| Silt | 4 | 0.701 | 21 | 218 | 3.1 | 35.4 | 0.0 |
| | 8 | 0.719 | 27 | 219 | 4.1 | 34.3 | 0.0 |
| Kaolinite | 8 | 0.779 | 35 | 220 | 2.3 | 29.3 | 14.6 |
| Smectite | 4 | 0.964 | 45 | 220 | 0.9 | 17.9 | 49.4 |

**Modifications to the Dye Test to Alter the Adsorption of Clays**

The approach taken in attempting to improve the dye test took the form of various chemical treatments of the clays, combined with the use of different dyes. The use of different dyes was investigated because it had been suggested that a larger dye molecule would have less access to adsorption sites than a small dye molecule (1,3). X-ray diffraction analysis uses ethylene glycol to identify swelling clay minerals by expanding their inter-layer lattice structure; various glycols were investigated as treatments for the clays in the hope that they would preferentially target the expandable clay minerals and block access to internal dye adsorption sites.

These tests were conducted on oven-dried test portions of 10 g of kaolinite and 1 g of smectite in 100 mL of solution in a 250 mL conical flask. The concentration of the glycol treatment was changed by dilution with water. The clay suspension was mixed using a magnetic stirrer. The dye test was carried out using 1 and 2 mL aliquots of dye, and the end-point was determined using the spot-test method. Addition of dye was followed by 1 min. of mixing, after which the mixer was stopped and a pipette was used to remove a drop of the fines/dye suspension for the spot-test.

This procedure was continued until the halo appeared. The halo had to be maintained for 5 min. before the end-point was confirmed. If the halo disappeared at Minutes 2, 3, or 4, another 2 mL aliquot of dye was added, followed by mixing. If the halo disappeared after 5 min. of mixing, a 1 mL aliquot of dye was added, followed by the same mixing procedure. Where clay treatments resulted in significant reductions in dye adsorption, 1 mL and 0.5 mL aliquots of dye were used.

**Materials**

The cement used was Rochester Ordinary Portland Cement to BS12 (5). The clay minerals used were Laporte Absorbents, Mineral Colloid montmorillonite (smectite) and Remblend china clay (kaolinite). The clay minerals were dried at 105°C for 24 h before use. Whatman 40 grade filter paper was used for conducting the spot-tests. The sand used in the mortar

TABLE 4
Volumes of dye required to "neutralise" added clays and clay in sand fines/silt.

| Mix | Mass of dye g(dye) | Volume (mL) at 13.4 g(dye)/L |
|---|---|---|
| Sand | 2.4 | 178 |
| Sand + 1% silt | 2.4 | 178 |
| Sand + 2% silt | 2.4 | 178 |
| Sand + 1% kaolinite | 2.6 | 193 |
| Sand + 2% kaolinite | 2.7 | 200 |
| Sand + 1% smectite | 5.0 | 370 |
| Sand + 2% smectite | 7.6 | 565 |

mixes (and which was ground to produce non-clay fines for the control mixes) was a washed concreting sand to BS882, grade M (6) with a methylene blue value of 0.6 g(dye)/kg(sand). The dyes used are listed in Table 2. The glycols used in the modified dye tests were standard laboratory reagents as follows:

| | |
|---|---|
| Propane-1,2,3-triol (Glycerol) | $CH_2(OH).CH(OH).CH_2(OH)$ |
| Ethanediol (Ethylene glycol) | $CH_2(OH).CH_2(OH)$ |
| Di-ethylene glycol | $CH_2(OH).CH_2.O.CH_2.CH_2(OH)$ |
| Propane-1,3-diol | $CH_2(OH).CH_2.CH_2(OH)$ |
| Butane-1,4-diol | $CH_2(OH).CH_2.CH_2.CH_2(OH)$ |

## Results

### Mortars Made to Constant Workability

Table 3 presents the properties of mortars made to constant workability, initially judged using the subjective opinion of the technician and then repeated using the flow table.

### Mortar Made to Constant Water:Cement Ratio and Also Using Methylene Blue Dye

The quantity of methylene blue dye that would be adsorbed by the clay present in the sand and the added clay was calculated. A solution of methylene blue dye was prepared and calibrated against standard kaolinite and the volume of dye to be added as a replacement of the mix-water was calculated. The masses and volumes of dye are presented in Table 4. Table 5 shows the properties of mortar made to constant water: cement ratio.

### Modifications to the Dye Test

The purity of the methylene blue dye and the concentration of the cartasol dye liquids used are shown in Table 2 together with the quantities used to make 1 L of dye. The exact

TABLE 5

Properties of mortar made to constant water:cement ratio.

| Fines added as % mass of cement | | Water:cement ratio | Plunger mm | Flow mm spread | Air content % | 28 day $N/mm^2$ |
|---|---|---|---|---|---|---|
| Mixes made without methylene blue dye | | | | | | |
| None | 0 | 0.754 | 27 | 210 | 3.3 | 33.1 |
| Silt | 1 | 0.754 | 27 | 210 | 3.4 | 33.8 |
| | 2 | 0.754 | 27 | 197 | 3.1 | 34.3 |
| Kaolinite | 1 | 0.754 | 28 | 200 | 3.4 | 33.7 |
| | 2 | 0.754 | 30 | 205 | 2.9 | 34.5 |
| Smectite | 1 | 0.754 | 21 | 185 | 3.0 | 34.2 |
| | 2 | 0.754 | 18 | 170 | 3.2 | 32.2 |
| Mixes made with methylene blue dye added to mix-water to cancel effect of clays | | | | | | |
| None | 0 | 0.754 | 41 | 240 | 4.0 | 30.1 |
| Silt | 1 | 0.754 | 38 | 225 | 4.9 | 30.1 |
| | 2 | 0.754 | 38 | 225 | 4.8 | 30.4 |
| Kaolinite | 1 | 0.754 | 41 | 230 | 4.3 | 31.3 |
| | 2 | 0.754 | 42 | 230 | 4.3 | 31.0 |
| Smectite | 1 | 0.754 | 40 | 220 | 5.1 | 31.7 |
| | 2 | 0.754 | 42 | 225 | 5.7 | 31.7 |

concentrations of the cartasol dye liquids were not known and were estimated on the basis of the limited data obtained from the manufacturer. Based on the work of Pike (4), between 114 g and 119 g of the concentrate was used to make 1 L of dye. At a concentration of 9% for cartasol red dye and 12.5% for cartasol blue and cartasol turquoise dyes, this gave actual dye concentrations ranging from 10.3 to 14.9 g/L. These values were used to calculate the quantities of dye adsorbed per kg of kaolinite. For example, kaolinite adsorbed 12 mL of cartasol red per 10 g of clay, equating to 12.4 g(dye)/kg clay. These figures are compared with those obtained by Pike (Table 6). Results are expressed as quantities of dye adsorbed and as a ratio of adsorption, kaolinite:smectite (K:S).

TABLE 6

Ratios of dye adsorptions for kaolinite and smectite using cartasol dyes.

| | Yool and Lees | | Pike | |
|---|---|---|---|---|
| Dye | g(dye)/kg K | K:S ratio | g(dye)/kg K | K:S ratio |
| Cartasol Red | 12.4 | 1:28 | 12 | 1:28 |
| Cartasol Turquoise | 8.9 | 1:37 | 9.2 | 1:26 |
| Cartasol Blue | 7.4 | 1:34 | 5.0 | 1:34 |

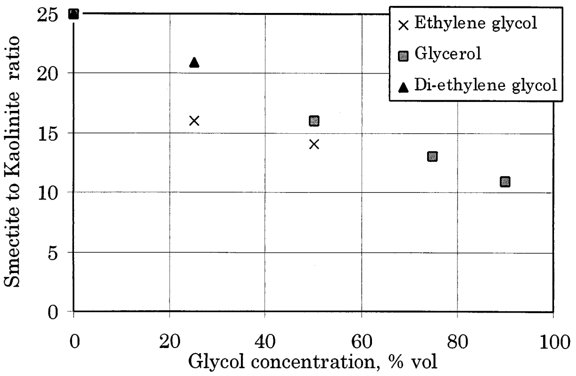


FIG. 1.
Effect of ethylene glycol, glycerol, and di-ethylene glycol on clay adsorption ratios using methylene blue dye.

## Use of Glycols and Four Different Dyes

The results obtained using four different dyes on two clays treated with three different glycols are summarised in Figures 1 to 3. Figure 4 shows the relative effect of ethylene glycol on the adsorption of cartasol red by kaolinite and smectite. The initial tests were carried out using methylene blue dye and glycerol, and although reductions in the adsorption of dye by the smectites were observed, difficulties were experienced in identifying the end-point. Glycerol is a viscous fluid that makes the characteristic blue halo of the end-point very indistinct and slow to appear.

Dilution of glycerol with water improved the halo formation at end-point but reduced the effect of glycerol on the adsorption of dye by the clays. The lowest ratio of adsorption for

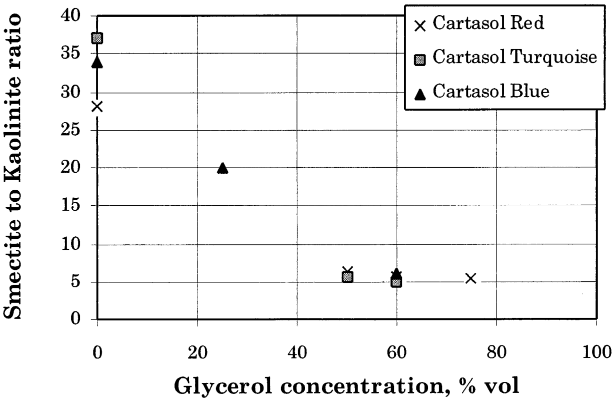


FIG. 2.
Effect of glycerol on clay adsorption ratios using cartasol dyes.

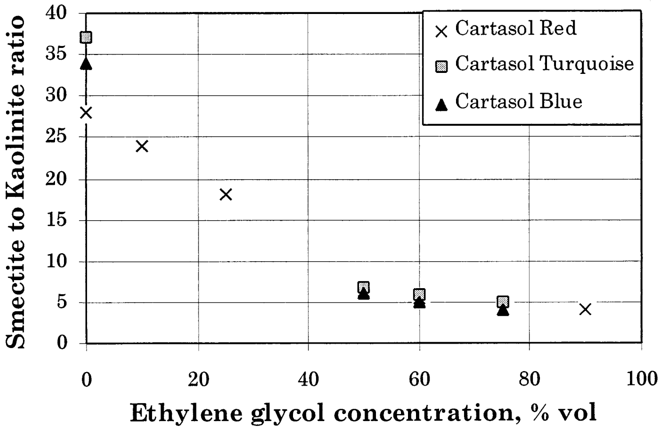


FIG. 3.
Effect of ethylene glycol on clay adsorption ratios using cartasol dyes.

kaolinite to smectite that could be achieved was 1:11. The tests using methylene blue dye were repeated using two of the other four treatments (ethylene glycol and di-ethylene glycol), but neither of these resulted in significant improvements on the results obtained with glycerol. The glycerol, ethylene glycol, and di-ethylene glycol were used at various dilutions with the other dyes. Dramatic reductions in adsorption by smectite were noted with all three of the cartasol dyes. With ethylene glycol at 75% by volume concentration with water and using cartasol blue dye, the ratio of dye adsorption for kaolinite and smectite was reduced from 1:34 to 1:4.

As well as reducing the ratio of adsorption for the different clays, the ethylene glycol was less viscous than glycerol, clarifying end-point determination. In addition, the cartasol blue

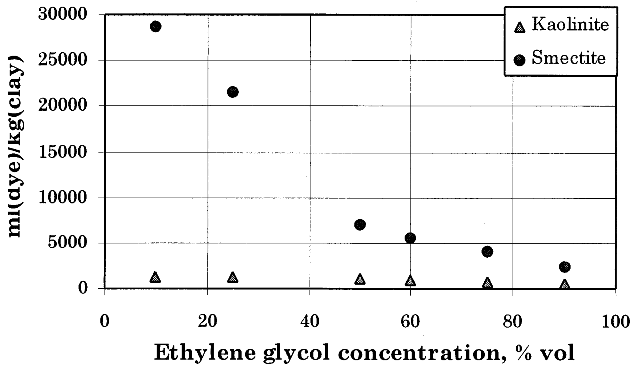


FIG. 4.
Effect of ethylene glycol on the quantity of cartasol red dye adsorbed by kaolinite and smectite.

dye produced a strong, dark blue halo at end-point, more distinctive than the lighter coloured cartasol red, cartasol turquoise, and methylene blue dyes. Several other glycols were tried but none gave satisfactory results, the full results of all the glycol treatments are not reported here.

## Discussion

### Mortar Mixes

The aim of the work on mortars was to confirm the work of other authors and, by using methylene blue dye to neutralise the clays, investigate the mechanism by which clays affect concrete and mortar. The results of this limited body of work confirm the relative effect of kaolinite and smectite on strength loss in mortar. The results, corrected to 1% addition of clay by mass of cement, compared to the relevant controls, show a ratio of effect of 1:4 kaolinite:smectite for mixes made to constant workability judged by eye and 1:7 kaolinite: smectite for mixes made to constant workability as judged by flow table. These values are generally consistent with the values quoted by other authors (3,4), particularly when the water:cement ratio is so critical to strength and the difficulties in defining, measuring, and obtaining a constant workability are taken into consideration.

However, this work has shown that the actual reductions in strength, for a given clay content, appear to be approximately twice those stated by other authors. 1% of kaolinite by mass of cement causes a 2% drop in strength (not a 1% drop in strength), and 1% of smectite by mass of cement causes a 10% drop in strength (not a 4% drop in strength).

The mortars made with sands dosed with methylene blue dye produced some of the most interesting results. Notwithstanding the changes to air content (the methylene blue dye appeared to have some minor air entraining properties), the change in workability of mortars made with smectite and to constant water:cement ratio was not observed in otherwise identical mortars dosed with methylene blue dye.

Compared to the non-dyed mixes, the mixes dosed with methylene blue dye showed a reduction in 28-day compressive strength, possibly due to the clays being harmful not just in the way that they affect water demand but possibly in some other way, e.g., by coating aggregate particles and weakening aggregate-cement paste bond. However, it can be seen from the results in Table 5 that if the methylene blue dyed mixes were repeated but to the same workability as the non-dyed control mixes, the water:cement ratio would have been reduced to obtain the required workability, presumably resulting in an increase in 28-day compressive strength.

It is suggested that the methylene blue dye occupied adsorption sites on the clays that would otherwise have been available to water. Not being bound in adsorptive clay, the water was then free to lubricate the cement and aggregate and contribute to workability.

### Improvements to the Dye Test

The most effective conditions for selectively reducing the dye adsorption of the smectites involved the use of dye molecules with molecular weights greater than that of methylene blue dye. This may be coincidental, because the other dyes used were all of the cartasol family and

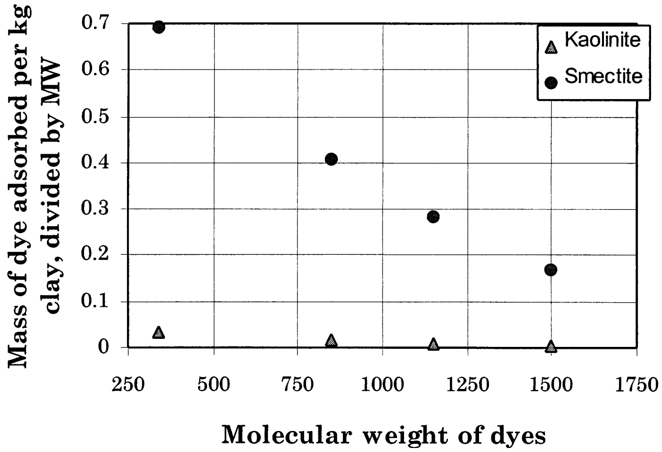


FIG. 5.
Effect of dye molecule size on dye adsorption.

all the ratios of adsorption for these dyes by untreated clays were greater than the 1:20-to-25 kaolinite:smectite ratios obtained using methylene blue dye.

However, when the results for untreated clays are expressed as the mass of dye adsorbed per kg of clay divided by the molecular weight of the dye (in order to reflect the differences in the size of the dye molecules), the adsorption of the smectites and the kaolinites decreased in line with increasing molecular weight (Fig. 5).

This model for dye adsorption is based on the assumption that molecular weight correlates with molecular size and that dye molecule size is a controlling factor in the adsorption of dye. The composition of the cartasol dyes is unknown and so cannot be compared with the composition of the methylene blue dye; this merits further investigation. Furthermore, it has been assumed that all the dyes are in a monomer state (methylene blue dye can exist as a monomer-dimer combination at certain concentrations).

At present, the difference in the quantities of the different dyes adsorbed can best be explained by assuming that the mass of dye adsorbed is a function of the molecular weight. However, the difference in adsorption behaviour between untreated clays and those treated with ethylene glycol would suggest that the methylene blue dye does not behave in the same way as the cartasol dyes. It is tentatively suggested that above a certain molecular weight, the adsorption of dye by smectites is restricted to external surfaces of the clay by the presence of a glycol (if present in sufficient concentrations) in the inter-layer spaces.

Finding such dramatic changes in dye adsorption dependent on glycol dilution or clay/glycol concentration was unexpected. If the glycol molecules inhabit the inter-layer sites of the smectites (where they could be expected to have a large effect), but have relatively little effect on kaolinite (with no inter-layer sites to soak up glycols in preference to dye) then why should glycol concentration have any effect on adsorption? It might be expected that even a relatively small quantity of glycol would be sufficient to block inter-layer adsorption sites. This latter observation would suggest that the water, glycol, and dye all compete to occupy "sites" in or on the clays and that their relative concentrations will dictate the final equilib-

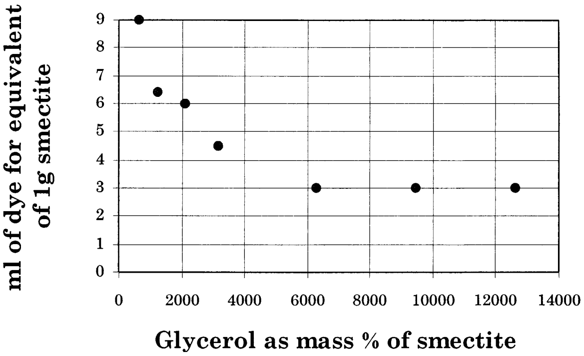


FIG. 6.

Cartasol blue adsorption by smectite at a range of clay to glycerol concentrations and at 50% by volume glycerol.

rium reached. Furthermore, tests using different concentrations of glycerol to clay at a given dilution (50% by volume) showed what appeared to be a minimum concentration of glycerol to clay below which adsorption of dye gradually increased (Fig. 6). There was a similar correlation for ethylene glycol and cartasol blue where doubling the mass of smectite for the same volume of ethylene glycol resulted in a tripling of the quantity of dye adsorbed.

## Conclusions

Kaolinite and smectite are associated with reductions in compressive strength of mortars in the ratio of ca. 1:5. For mortars made to the same workability, smectite causes a decrease in compressive strength five times that caused by an equal addition of kaolinite. This is similar to results reported by other authors. Kaolinite causes reductions in compressive strength of the order of 2% for a 1% addition of clay by mass of cement. Smectite causes reductions in compressive strength of the order of 10% for a 1% addition of clay by mass of cement. These reductions in strength are roughly double the results obtained by other authors.

Methylene blue dye has been used to “neutralise” the adsorptive properties of clays in aggregates used to make mortar and has restored workability to mortars containing harmful clay fines made to constant water: cement ratios.

Both methylene blue dye and the cartasol dyes used in titration tests for clay fines grossly overestimate the damaging effects of the smectites. Modifications to the dye test, that rank dye test results in line with clay harmfulness, target the inter-layer adsorption sites of the smectites and leave the kaolinites relatively unaffected. Glycols, notably ethylene glycol, significantly reduce the adsorption of dye by smectites. This effect is most notable with the cartasol dyes: cartasol red, blue, and turquoise. The effect of glycols on adsorption of dye by clay is concentration dependent. Both the degree of dilution of the glycol with water and the concentration of the glycol relative to the clay have an effect on dye adsorption. A combination of 6% ethylene glycol and the use of cartasol blue dye can reduce the ratio of dye adsorption for kaolinite to smectite from 1:34 to 1:5. This combination also has practical advantages in terms of performing a dye adsorption test, notably that acceptable test conditions prevail for obtaining a distinct halo at end-point. Adsorption of dye by clays appears to be related to molecular weight, the implication being that access to inter-layer adsorption sites is restricted for larger molecules. A model suggested for this system is that of competition between dye, water, and glycol molecules for access to "sites" within the clay.

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