

Efflorescence Control in Grout with MetaMax[®] EF

Evaluation of MetaMax[®] EF in Grout Formulation

Summary:

Efflorescence in cold weather applications can be significantly reduced with the addition of MetaMax[®] EF highly reactive metakaolin. By replacing a percentage of cement with MetaMax[®] EF and reducing the amount of thickener in the grout formulation, efflorescence can be controlled without compromising workability.

MetaMax [®] EF Level	Thickener Level	Efflorescence Rating @ 96 hours
0% - Control	0.05%	Severe
5%	0.03%	Slight
8%	0.03%	Very Slight
10%	0.02%	None to Very slight

Control at 96 hours, Severe efflorescence



5% MetaMax[®] EF @96 hours, Slight efflorescence



8% MetaMax[®] EF @96 hours, Very slight efflorescence



10% MetaMax[®] EF @96 hours, None to Very slight efflorescence



Background:

Grout products have the tendency to exhibit severe efflorescence during cold weather applications. In application below 50 F efflorescence was apparent and uncontrollable.

Objective:

The scope of this study was to adjust a standard grout formulation that will allow the addition of MetaMax[®] EF in order to help reduce efflorescence in cold weather applications without compromising workability.

Experimental:

This study evaluated three cementitious mixtures at increasing levels, 5%, 8%, & 10%, of MetaMax[®] EF and was compared to a control mixture. Thickener levels also were adjusted to achieve adequate workability level.

Tests conducted included workability, appearance of grout at room condition in contact with saltillo and porcelain tiles, and appearance of grout at low temperature between quarry tiles.

Mixtures:

Cementitious material: Cementitious materials were achieved by replacing cement, with MetaMax[®] EF (5%, 8%, & 10% replacement levels)

Workability controls: (two were used)

Control #1: standard mixture at a ratio of 72% Base Material / 28% white cement with 0.05% thickener based on total dry batch weight, 20% water.

Control #2: Commercially available grout product.

Workability Test: A ratio of 72% Base Material / 28% cementitious material at 10% level of MetaMax[®] EF with varying levels of thickener based on total dry batch weight.

Appearance Control: mixture at a ratio of 72% Base Material / 28% white cement with 0.05% thickener based on total dry batch weight.

Appearance Tests: A ratio of 72% Base Material / 28% cementitious material at 5, 8, & 10% levels with the thickener level determined during the workability testing, based on dry batch weight.

Mixing Methodology:

A Kitchen Aid mixer with a wire whisk attachment was used to mix all dry materials. All dry materials were blended together before adding to mix water.

Cementitious material was produced first by mixing cement with MetaMax[®] EF and blending for 3 minutes at low speed to achieve a uniform mixture. Base material and thickener were then added and blended at low speed for an additional 3 minutes; this was time enough to achieve uniformity.

After all dry materials were well blended; the mixture was then added to the mix water. A Hobart brand commercial mixer, with a spade attachment, was used for mixing the grout. The mixer was run at low speed for 30 seconds, stopped in order to scrape down the sides of mixing bowl, then ran for an additional 30 seconds at low speed. Grout was then allowed to slake for 10 minutes and then re-mixed using a spatula.

This methodology was used throughout the study.

Testing and Storage Conditions:

The mixing room conditions during this study were 70°F temperature and 58 % RH. Workability and initial appearance testing were done under these same conditions. Mix water temperature was constant at 70°F. Low temperature sample storage was at 42°F temperature and 38 % RH.

Workability Testing:

First challenge was to gain a hand on the workability of the material. With very little experience in working with grout I had no feel for the workability of the product. ASTM has two standards that relate to the workability of hydraulic cement, ASTM C 230 (Standard Specification), and ASTM C1437 (Test Method). These two standards were adopted for testing grout workability. It is emphasized that these tests were not developed for testing grout and the data produced from this test should be only used for comparison of a test material to a control material. The standard deviation of ten tests ran with a

commercially available product was around 8%, this is about twice the conclusion of ASTM for the bias of one technician in one lab under same conditions.

Workability control mixture #1 was produced by blending Base material batch at 72%, white cement at 28%, and thickener at 0.05% of dry batch weight. Grout was made with 20% w/cm ratio, which achieved a flow of 102%. Compared to control mixture #2, a commercially available material which was also made with 20% w/cm ratio, achieved a flow of 84%

A workability test mixture was produced by blending a ratio of 72% Base Material / 28% cementitious material at a 10% MetaMax[®] EF level with varying levels of thickener based on total dry batch weight. The grout from each of the mixes was produced and tested at 20% and 18% water levels.

Thickener Level	20% Water Content	18% Water Content
0.05%	89%	78%
0.03%	90%	81%
0.02%	94%	88%
0%	121%	109%

The cementitious material at the 10% MetaMax[®] EF level with 0.02% thickener achieved a flow that is near the median of the two workability control mixtures. The 5% & 8% cementitious materials required the addition of 0.01% thickener to achieve a comparable flow. These are the formulations that were chosen to complete the appearance testing.

**Note that the mixture containing the MetaMax[®] EF, both at the 18% and 20% water levels appeared to be somewhat *stiffer* than the control after the slaking period. Light re-mixing brought the material back to a workable state without the use of additional water. All workability testing was conducted immediately following this re-mixing.

Appearance Testing:

The first appearance test that was conducted was to place the grout mixture in a template cut to resemble grout lines. The template was placed so that the grout would cure in contact with a tile. Saltillo and porcelain tiles were used for this test. The grout was allowed to cure for 28 hours at room temperature, and then removed from the template for evaluation.

There was a slight difference in the appearance between the grout that was cured on the Saltillo tile to the grout that was cured on the porcelain tile. The grout cured on the saltillo tile appeared to be drier and slightly lighter in shade. Color saturation, however, seemed to be fairly uniform throughout both samples.

The slight difference seen with the control mix between the two types of tile was also noted in the test mixtures that were cured in the same manner. In comparing the test mixtures to the control cured on the same type of tile very little difference was noted

The second appearance test that was conducted involved placing grout between quarry tiles that were set to concrete backer board with an adhesive mortar. These tile plates were allowed to cure for 24 hours then placed in low temperature storage for an additional 16–18 hours to adjust to the low temperature conditions. The plates were then removed and grouted within 5 minutes after removal. Once grouted the plate was returned to the low temperature storage. After 45 minutes, the tiles were cleaned with sponge in a circular motion to shape grout, and then tiles were allowed to cure for 24 hours. After 24 hours the tiles were again cleaned and returned to low temperature storage.

Photographs and notes of appearance were taken every 24 hours for the first 96 hours. (See below) Plates remained in low temperature storage for a total of 7 days and last evaluated upon removal.

Control Mixture

24 hour: after cleaning, appearance of grout was smooth and even.

48 hours: strong color variations are noted, possible efflorescence noted in areas but very slight.

72 hours: efflorescence was noted in the majority of the grout line, color variations had decreased or were being covered by the efflorescence.

96 hours: efflorescence had completely covered the grout lines between the tiles.

7 days: little change from 96 hours, entire grout line appears to be white with efflorescence.

5% Cementitious Mixture

24 hour: after cleaning, appearance of grout was smooth and even.

48 hours: color variations are noted, no efflorescence noted.

72 hours: color variations continue, slight efflorescence noted just on the edge of tiles.

96 hours: color variations continue, slightly more efflorescence noted in some areas along the edges of tiles.

7 Days: color variation continues, slight efflorescence is now noted in center of grout line, compared to the control there is a significant reduction of efflorescence.

8% cementitious Mixture

24 hour: after cleaning, appearance of grout was smooth and even.

48 hours: slight color variations are noted, no efflorescence noted.

72 hours: slight efflorescence noted along the edge of some tiles.

96 hours: curing continues, no increases in the amount of efflorescence

7 days: just slightly more efflorescence noted. Compared to the 5% cementitious material there is a again a reduction in efflorescence.

10% Cementitious Mixture

24 hour: after cleaning, appearance of grout was smooth and even.

48 hours: no efflorescence noted appears to be curing normally.

72 hours: still no efflorescence noted, very slight lightens of grout along the edge of some tiles.

96 hours: curing continues, very slight amount of efflorescence noted just along the edge of some tiles.

7 days: no increase in efflorescence noted

Research Conducted by:

Charles E. Cleary

ACI Certified Concrete Laboratory Testing Technician

Engelhard Concrete Laboratory

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