

EFFECTIVNESS OF INORGANIC CONCRETE COATING MATRICES  
AND THEIR SELF CLEANING PROPERTIES

by

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## **ABSTRACT OF THE THESIS**

### **EFFECTIVNESS OF INORGANIC CONCRETE COATING MATRICES AND THEIR SELF CLEANING PROPERTIES**

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Dr. P. Balaguru

Concrete Coatings are becoming more popular to protect concrete surfaces for infrastructures. The coating improves the durability of concrete structures. Inorganic concrete coatings are better than organic coatings because they allow the release of vapor pressure. The results presented in this thesis deals with the behavior of the inorganic matrices which are based on silicates. The coating is compatible with concrete, brick and wooden surfaces.

The objective of the research presented in this thesis is to evaluate the matrices for workability, ease of application and self cleaning properties. Cracking characteristics were also evaluated using high magnification. The self cleaning properties of the coating matrices were studied using Rohdamine B dye to detect the degradation rate of the organic chemicals that represent the organic pollutants in the atmosphere.

Advanced technology such as X-ray photoelectron spectroscopy and Atomic force microscope was used to measure the curing period and its effect on the matrices self cleaning properties. The degradation of the organic chemicals represents a breakthrough in the development of the inorganic matrices because they can be used in many applications. Matrix combination that provides workable mix and minimum nano-level cracking are recommended.

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## **CHAPTER 1. INTRODUCTION**

### **1.1 Scope and Objective of this Study**

At the present time, concrete is being used in every aspect of human civilization. Initially, use of concrete was limited only to building construction, but nowadays concrete applications are quite common in various industrial sectors. For example, fertilizer, petrochemical, refinery, water and waste water treatment facilities, heavy chemical industries, highways, bridges...etc. Due to huge corrosion on iron structures, the industries as a whole are moving towards concrete coating over the existing structures. What are the best ways to develop and maintain the concrete structures surface? How can we create an ultimate concrete coating that is safe, durable and efficient? Is it possible or practical to make a concrete paint that is self cleaning and has good workability? This research program is to give comprehensive answers for these questions through series of experiments that have been performed on different concrete mixes. The Ultimate concrete coating presented in this thesis is a series of mixes that will have many properties like self cleaning properties, graffiti proof, crack control and good workability. In addition, it can be used in the rehabilitation of deteriorated concrete surfaces; which is a major problem as it leads to structural problems and reinforcement corrosion. This research program will also try to analyze some specific problems that took place during the study of the concrete mixes, from its beginnings to its development.



## CHAPTER 2. LITERATURE REVIEW

### 2.1 Introduction and historical background:

Before discussing the areas in which concrete coating must be used, the purpose and the history of concrete coatings should be identified. Concrete coating is a barrier coating which is applied on the concrete structures surface to ensure considerable durability and give further protection against penetration of carbon dioxide, water and other aggressive chemicals that can have a significant effect on the behavior of these concrete structures. The intrusion of water and other chemicals is the major reason for deterioration of concrete structures. For the plain concrete, the failure occurs by the exposure and popping of aggregates. However, for reinforced concrete, steel reinforcement corrosion is the main reason for the deterioration of concrete. Reinforcement corrosion accelerates the degradation process by creating more cracks and further chemical access.

Any history of concrete coating usually starts with the famous ancient Egyptian Temples and cave paintings of Europe. Starting the mid to late 1800s, paint was usually made by professional painters, craftsmen who both prepared paint and applied it. In that time, the majority of the paint was prepared on site. Interior surfaces were often coated with whitewash, distempers or casein paints which had either no binder at all, or binders such as animal glue or casein which had poor durability. Casein paints remained popular into the 20th century, purchased as powders to be mixed with water just before use & some are still available commercially (Vanketsh, 1999). Exterior paints were made by mixing white lead paste into oil. Lead functioned both as white pigment and as drying

agent for curing of the oil. The industrial revolution brought about centralized manufacturing of paint dispersions as well as the production of a variety of resin technologies. The first appearance for ready mixed paints in the US was granted in 1867. Sales began in earnest in the mid 1880s and paint factories started. By 1900 nearly 20 million gallons per year of ready mixed paint were sold in the US, and this volume rose steadily. By 1930, lithopone and zinc oxide had mostly replaced lead for interior paints; however, lead was still used widely for exterior paints and metal coatings.

The first titanium dioxide pigments were introduced in the 1930s. The early versions of titanium dioxide were mainly in crystal form. Exterior paints for a time were intentionally formulated with a  $\text{TiO}_2$  as self cleaning paints for a small amount of chalking which allows dirt to be removed by rain. Titanium dioxide was commercialized after World War II and finally allowed the formulation of durable paints that do not contain lead (Vanketsh, 1999).

## **2.2 Characterization of the inorganic coating matrix:**

Portland cement is the most widely used inorganic binder in the civil industry. On the other hand, the size of the Portland cement grains is one of the major disadvantages of the Portland cement system because it is relatively large. Thus, it prevents the formation of thin binders. Other common room temperature matrices such as alluminosilicates and phosphate based compounds were introduced as an alternative to Portland cement. One of the major advantages of the silicate compounds is that they are not 100% impermeable thus allowing the concrete surface to breath or in other words to release the vapor pressure. In addition, they are not toxic and they have proven to be UV light resistant and fire resistant.

One of the inorganic resins which are currently available is a potassium alluminosilicate, or poly (sialate-siloxo), with the general chemical structure.



There are many research programs and studies that have been conducted on the performance of inorganic polymers as a protective coating. Protective concrete coating can be simply described as the coating that protects the surface from the intrusion of water and other chemicals that cause deterioration and failure of concrete structures.

These coatings act as a barrier that prevents the ingress of liquids and other chemicals but at the same time they do not allow the concrete to breathe or in other words they do not allow the release of water that is already inside the concrete. Accumulation of water at the interface eventually led to the peeling of the organic coatings (Balaguru, 1998). The results reported in Balaguru and Nazier, 2004, deal with an experimental study on the performance of the inorganic concrete coating. The results of the research program shows that the inorganic coating is resistant to UV light, releases vapor pressure and is fire resistant. Moreover, the inorganic coating is durable under scaling, wet-dry and freeze-thaw conditions (Nazier, 2004 ).

### **2.3 Evaluation of the inorganic coating matrix:**

There are many studies that discuss many means of concrete coating evaluation.

However, all these studies has took in consideration three important factors which are :

- 1) Coating Workability
- 2) Ease of application
- 3) Cracks development

In order to understand the basis of for evaluation of the inorganic matrix, those points have to be identified and clarified.

### **2.3.1 Workability:**

Workability is the term used to describe the property of concrete coating that determines the ease with which it can be mixed, applied, and finished to a homogenous condition (ACI 116R-00, 73). The homogeneity and the consistency of the coating mix is very important for the application. The mix can have low viscosity and yet it is not workable. For example, there are lots of mixes that form lumps or in case of very thin mixes or in other words very low viscosity mixes.

### **2.3.2 Ease of application:**

There are many techniques to apply the concrete coating. The most widely used techniques are brush, roller, and spraying. A large number of commercial brushes, rollers and sprayers are available in the market. For the inorganic binders, the preferred methods are by brush or rollers because this technique provides better wetting. Ease of application is directly proportional to workability. Thus, the more workable the coating, the easier it can be applied.

### **2.3.3 Cracks development:**

Concrete coatings crack for many reasons. Shrinkage is the primary cause of cracking development. It is a very well known fact that as concrete coating hardens and dries, it shrinks. This is due to the loss of excess mixing fluids such as chemical admixtures or water. Thus, in most cases, the wetter or soupier the coating mix, the greater the shrinkage will be. This shrinkage causes forces in the mix which literally pull the coating particles apart and cracks are the end result of these forces. One of the main reasons of

cracks development is the surface properties. Surface roughness is directly proportional to cracks development. Thus, surface preparation is very important to minimize the cracks.

#### **2.4 Surface Preparation:**

Surface preparation and treatment of concrete prior to coating application is one of the most important keys for the coating adhesion and surface protection. Surface preparation can be determined by several factors such as type of concrete and its compression strength, age of concrete, concrete placement, concrete curing and finishing processes, previous contamination of the concrete (which can occur due to exposure to chemicals, salinity... etc) and concrete conditions, like bug holes, reinforcement corrosion, exposed aggregate, & rebar corrosion. Surface preparation is very important especially in the case of organic binders. However, inorganic binders don not need lots of surface preparation as they can be applied on a wet surface. In addition, the surface needs not to be totally dust free.

Special equipment has been developed for sand blasting, cleaning, automatic metering and application on horizontal surfaces, but this heavy equipment is not appropriate for the use on vertical surfaces (Garon, 2000). The fact that inorganic binders can be applied on rough surfaces does not neglect the significance of surface preparation on cracks development. Hence, surface preparation is very important for surface roughness and the minimization of cracks.

## CHAPTER 3. EXPERIMENTAL DESIGN

### **3.1 Introduction**

Geopolymers are inorganic polymers widely used in many applications for more than thirty five years. Geopolymer was first applied to these materials by Joseph Davidovits in the 1970s, although similar materials had been developed in the former Soviet Union since the 1950s under the name Soil Cements. Davidovits proposed in 1978 that a single aluminum and silicon-containing compound, most likely geological in origin, could react in a polymerization process with an alkaline solution. The binders created were termed "geopolymers" (Wikipedia, 2004). They are an attractive replacement to cement as they have lower carbon dioxide emissions. Geopolymers can be produced using residual waste products such as fly ash and red mud. Geopolymer based material has many advantages such as high chemical and thermal resistance at both atmospheric and extreme conditions. One of the geopolymers applications is coatings and paintings.

In this chapter, geopolymers based concrete coatings are being discussed in detail. Fifty mixes were prepared and the components were introduced in a form of a matrix named "Ultimate Concrete Coating Matrix". Those mixes were evaluated in terms of workability, ease of application and cracks development. Using advanced technology, color pigments were added to the mix giving it some glowing properties. This coat can now be used not only as a protection coat but also for traffic signals and night applications.

### **3.2 Mix Components**

The Mixes of the concrete coating matrix is composed of the following:

- 1) Silicate solution (Part A)
- 2) Silicate powder (Part B)
- 3) Hardeners
- 4) Fillers
- 5) Titanium dioxide (TiO<sub>2</sub>)
- 6) Distilled Water (H<sub>2</sub>O).
- 7) Admixtures
- 8) Retarders
- 9) Glowing powder

The components are described briefly in the following sections.

#### **3.2.1 Silicate Solution**

Silicate solutions are extensively used in the painting and coating industry. They play an important role in the chemical industry and they are used to manufacture many industrial as well as commercial goods and products, for example chemical reagents and neutralization of acids, further as basic standard in analytics and many more applications. Silicate solutions are based on high single or mixed silicon dioxide to alkali metal oxide mol ratio inorganic alkali metal silicates. Silicate solution is produced from a starter alkali metal silicate aqueous solution by mixing the solution with a silicone monomer and agitating until hydrolysis is essentially complete. A silica gel is then added as an aqueous

slurry and blended until the silica gel is at least partially dissolved. Thereafter, the mixture is agitated to a smooth consistency and recovered as a binder composition (Brito, 1998). Silicate solution serves as a binder in the mix and it gives the mix higher workability and makes it easier to be applied on variety of surfaces.

### **3.2.2 Silicate Powder**

Silicate powder is mainly composed of mineral geopolymers and silicates byproducts which are considered as residuals from production of some silicon alloys. Geopolymer are created from aluminum silicate materials and they act as an alternative for Portland-based cements. Geopolymers are generally formed by reaction of an aluminum silicate powder with an alkaline silicate solution at roughly normal atmospheric conditions.

Fly ash is a commonly used material for the manufacturing of geopolymers, and is generated by thermal activation of coal residual in the generating plants.

The chemical reaction that takes place to form geopolymers follows a multi-step process which can be summarized in the following points (geopolymers institute, 2005):

- 1) Dissolution of Si and Al atoms from the source material due to hydroxide ions in solution
- 2) Reorientation of precursor ions in solution, and Setting through polycondensation reactions into an inorganic polymer.
- 3) The inorganic polymer network is in general a highly-coordinated 3-dimensional aluminum silicate gel, with the negative charges on tetrahedral Al(III) sites charge-balanced by alkali metal cations.



The ratio of Silicate: Aluminum in the polysilicate structure determines the mechanical properties of the geopolymers and their application fields. A low ratio Silicate: Aluminum initiates a three dimension Network that is very rigid. A high ratio Silicate: Aluminum provides polymeric character to the geopolymeric material. The silicate :Aluminum ratio is case sensitive because the coating should prevent the access such as salts and chlorides from accessing the surface and in the same time it should allow the concrete to breath. Otherwise, the coating will delaminate due to liquid collection at the interface (Balaguru, 2006). Silicate powder together with the silicate solution is the cementing agent that acts as a binder for the coating.

### **3.2.3 Hardeners**

A Hardener is substance or mixture added that is added to a the coating mix to take part in and promote or control the curing action, Also a substance added to control the degree of hardness of the cured coating. The hardeners used in the coating mix were mainly composed of metal oxides that are nearly insoluble in water but soluble in acids and alkalis. Many metal oxide pigments are used in the painting industry and also used in coatings for paper. Some Metal oxides particles absorbs both UVA and UVB rays of ultraviolet light, they can be used in ointments, creams, and lotions to protect against sunburn and other damage to the skin caused by ultraviolet light. Metal oxide mixture provide curing and hardening to the concrete coating at the room temperature.

### 3.2.4 Fillers

Fillers are something that fills the gaps or in other words the air voids in the coating mix giving it strength and helps to minimize the cracks. In the coating mix, fillers are mainly composed of fibers, metallic fibers and silicate compounds. Fibers are widely used in many applications such as fabrics and textile industry. Fibers are mainly created from polyester and polyamides. However, metallic fibers are composed of metal, plastic-coated metal, metal-coated plastic, or a core completely covered by metal. The mixture of fibers and silicate compounds gives strength to the mix and minimizes the cracks. In addition, it gives the coating thermal resistance properties which make the coating suitable for high temperature conditions.

### 3.2.5 Titanium Dioxide (TiO<sub>2</sub>)

Titanium dioxide is the naturally occurring oxide of titanium and it was first produced commercially in 1923. Its chemical formula is TiO<sub>2</sub> and when used as a pigment, it is called titanium white, Pigment White 6, or CI 77891 (Wikipedia, 2004). It is commonly used in many application applications that such as painting, sunscreen and food coloring. In 2004, 4.4 million of titanium dioxide tones were produced worldwide. Most titanium dioxide pigment is produced from titanium mineral concentrates by the chloride or sulfate process, either as the rutile or the anatase form (the crystalline structure of existing TiO<sub>2</sub> in the ores). The primary particles are typically between 0.2 and 0.3  $\mu\text{m}$  in diameter, although larger aggregates and agglomerates are formed. Ultrafine grades of titanium dioxide have a primary particle size of 10–50 nm and are used extensively as ultraviolet blockers in sunscreens and plastics, and in catalysts

(Henrich and Cox, 1994). Most commercial titanium dioxide products are coated with inorganic; for example alumina, zircon, silica, and organic; for example polyols, esters and silanes, compounds to control and improve surface properties. Titanium dioxide is also known to be photocatalyst that can break down almost any organic compound it get in contact with when exposed to UV light whether it was artificial light or sunlight. Nowadays, many products are developed that uses the photocatalytic action of titanium dioxide. These products include self-cleaning fabrics, paintings, and ceramic tiles. Moreover, titanium dioxide is used in the manufacture of paving stone that uses the catalytic properties of  $\text{TiO}_2$  to remove nitrogen oxide from the air, breaking it down into more environmentally basic substances. Hence, it plays in an important role in the degradation and decomposition of organic pollutants. Titanium was added to the coating mix to give it self cleaning properties which will be discussed in details in chapter 3.

### **3.2.6 Admixtures**

Admixtures are generally used as a solvent for many substances and they are used in the manufacture of many industries such as paintings, scents, colorings, and medicine. They are also used in the fuel industry for the internal combustion engines because they make fuel environmental friendly as it burns cleanly. Admixtures added to the mix are flammable, colorless, slightly toxic chemical compounds. Admixtures were added to the coating mix to improve surface smoothness and workability.

### **3.2.7 Retarders**

Retarder is the term used to express chemical agent that slows down a chemical reaction; thus, increasing the setting time and decreasing the strengthening rate during the early age. For example, the admixtures that is added to concrete mixes to slow down their chemical hardening. Retarders used in the coating mix were added to delay the chemical hardening of the coating mix without affecting the long-term mechanical properties so that it can be used in variety of applications.

### **3.2.8 Glowing Powder**

Glowing powder pigments was added to the coating mix to give the mix glowing properties at the dark; hence, it can be use for traffic signals and night applications. The mechanism of the glowing coating is that the glowing powder absorbs light and then releases it creating the glow in the dark effect.

## **3.3 Sample Preparation**

Three types of specimen were made. The first type of samples was made on concrete bricks 7.5 X 3.5 x 2.5 inch or circular samples of diameter 4 inch from commercially available concrete blocks. The circular samples were cut using a wet saw. The samples were let to dry for about 48 hours days. The second type of specimens was made of red bricks of 7.5 X 3.5 X 2.5 inch. After that the samples were cleaned using a piece of cloth to remove any lose particles. The third type of samples was made on James hardy board in the CAIT Materials laboratory in Livingston. The board surface was cleaned using piece of cloth and lose particles were removed. The area to be coated was

outlined using mask tape to achieve a perfect rectangle. Multiple mixes were made to cover the large area.

### **3.4 Preparation of Base mixes**

The Ultimate coating is prepared as follows. A mixture of 100 grams of silicate solution and 135 grams of silicate powder is placed in a high-shear mixer containing notched stainless steel blades and mixed for one minute at speed of 1,500 RPM. Few powder particles stick to the wall of the mixer. A putty knife was used to collect the particles sticking to the mixer wall and the mixture is mixed for 1 minute. A mixture of 10 grams of metal oxide, 15 grams of micro fibers, 5 grams of titanium dioxide and 3 grams of fibers is added to the mix and mixed for one minute. Putty knife is used again to collect all the mixture particles sticking to the mixer wall and mixed for one more minute. Ten grams of distilled water is added to the mix and mixed for one minute. Some problems appear during the mixing procedure for the silicon dioxide based mixes due to high shrinkage. The steel blades of the mixer were stopped by the formation of big sized patches as shown in

Fig 2.1. These problems were solved by adjusting the Silicate solution : silicate powder ratio.



**Fig. 3.1 shows the patch formation during the mixing procedure**

### **3.5 Preparation of Other mixes:**

#### **3.5.1 Retarder Mixes**

Same procedure of mixing as in the base mixes is used. The retarder is dissolved in distilled water then added to the mixture & mixed for one minute.

#### **3.5.2 Admixtures Mixes**

Same procedure of mixing as in the base mixes is used. Admixtures were added to the mixture and mixed for one minute. Then, distilled water is added to the mix and mixed for one more minute.

#### **3.5.3 Glowing Powder Mixes**

The glowing powder was added to the mixture during the second step and mixed for one minute. Then, distilled water is added to the mix and mixed for one minute.

### **3.6 Ultimate Coating Mix application**

Initially, Self cleaning coating mix is stiff and eventually mixes to a thick liquid that can be applied using brush or roller. The mix was applied on the three types of samples using a smooth brush or a roller. The samples were left at room temperature for at least 21 days before testing.

### **3.7 Curing Method**

The coating was cured at room temperature. At room temperature, the sample has to be protected from running water or direct rain for 3 days. After 24 hours, the samples are

water resistant. However, running water could damage the surface by leaching out small amounts of mix components.

### **3.8 Lab Investigation**

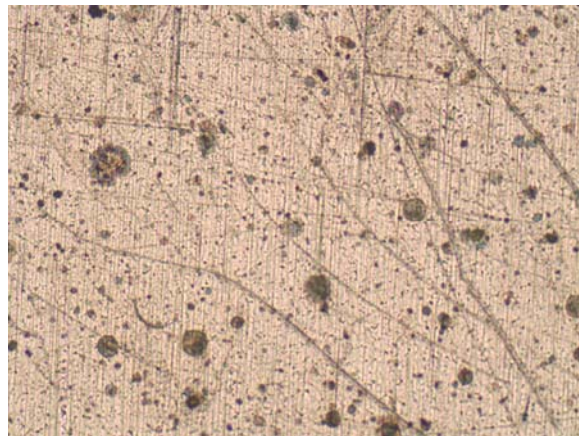
The performance of the inorganic coating was evaluated with time. Visual inspection along with digital microscopic photography will be used to evaluate the performance of the ultimate coatings. The microscope used for lab investigation is Olympus model GX41 and it is attached to a digital camera that is connected to a personal computer. Small samples were cut from the bricks using a wet saw so that the samples can fit the microscopic stage.



**Fig 3.2 shows the sample on the microscopic stage**

Samples made were checked under 50X lens (scale 500  $\mu\text{m}$ ) and 4X lens (scale 25  $\mu\text{m}$ ). The main goals of the lab investigation were to evaluate the coating mixes on the following Basis:

- 1) Workability
- 2) Ease of Application
- 3) Cracks Development



**Fig 3.3 shows the sample under 50X (500  $\mu\text{m}$ ) magnification lens**

The digital microscopic pictures taken under the 50X lens (500  $\mu\text{m}$ ) were similar to a great extent and the cracks was not shown clearly. Thus, 4X (25  $\mu\text{m}$  scale) lens was used to take the digital microscopic pictures and the cracks were identified. Each sample scanned under the microscope and a representative picture was taken that shows the main cracking spot or the main defects in the sample. The setting time for each mix was calculated by pouring small amount of each coating mix in a small plastic cup and the mix was checked every minute till hardening using a putty knife.



**Fig 2.4 shows the coating mix sample placed in a plastic cup to measure the setting time**



**Fig 2.5 shows the sample mix during the hardening time**



Table 3.1 and Table 3.2 give the main components of the samples coating mixes made in the lab with the different mixtures weights. The tables show the chemical composition of the different samples. Then, each sample mix was identified exclusively and three pictures were taken for the sample. The three pictures taken represent the following:

- 1) General layout of the sample.
- 2) A magnified picture was cut from the general layout picture using Adobe Photoshop software representing the main visual observation on the sample.
- 3) Digital microscopic picture under 4X lens (scale 25  $\mu\text{m}$ ) represents the main cracking spots or defects on the sample.

### **3.9 Sample Scoring scale basis**

Each sample was given a score ranging from 0 to 5 according to its workability, ease of application and cracks development. Score Zero means that sample has a very low workability and cannot be easily applied on the surface. Score five means that the sample has a very high workability and can be easily applied. For cracks development, Score Zero means that the sample has almost no cracks or cracks less than 1  $\mu\text{m}$  and score five means that the sample is full of cracks.

**Table 3.1 (Silica Powder 1)**

Mix No.	Components														Remarks
	A (gms)	B (gms)	C (gms)	Micro fiber (gms)	Ground Filler (gms)	TiO2 (gms)	Fibers (gms)	H2O (gms)	Retarder 1 (gms)	Admixture 1	Admixture 2 (Percentage)	Retarder2 (gms)	Glowing Powder (gms)		
Base Mixes															
1	100	125	10	15	30	10	3	10	-	-	-	-	-	Using Nano TiO2	
2	100	125	10	15	30	5	3	10	-	-	-	-	-	Using Coarse TiO2	
3	100	125	10	15	30	5	-	7.5	-	-	-	-	-	Using Nano TiO2	
Water Mixes															
4	100	125	10	15	30	-	3	10	-	-	-	-	-		
5	100	125	10	15	30	-	3	15	-	-	-	-	-		
6	100	125	10	15	30	-	3	20	-	-	-	-	-		
Retarder 1 Mixes															
7	100	125	10	15	30	-	3	10	0.25	-	-	-	-		
8	100	125	10	15	30	-	3	10	0.5	-	-	-	-		
9	100	125	10	15	30	-	3	10	1	-	-	-	-		
10	100	125	10	15	30	-	3	10	2	-	-	-	-		
11	100	125	10	15	30	-	3	10	4	-	-	-	-		
Admixture 1															
12	100	125	10	15	30	5	3	10	0.5	1	-	-	-		
13	100	125	10	15	30	5	3	10	0.5	2	-	-	-		
14	100	125	10	15	30	5	3	10	0.5	3	-	-	-		
15	100	125	10	15	30	5	3	10	0.5	4	-	-	-		
Admixture 2 Mixes															
16	100	125	10	15	30	5	3	10	0.5	-	1	-	-		
17	100	125	10	15	30	5	3	10	0.5	-	2	-	-		
18	100	125	10	15	30	5	3	10	0.5	-	3	-	-		
19	100	125	10	15	30	5	3	10	0.5	-	4	-	-		

Mix No.	Components														Remarks
	A (gms)	B (gms)	C (gms)	Micro fiber (gms)	Ground Filler (gms)	TiO2 (gms)	Fibers (gms)	H2O (gms)	Retarder 1 (gms)	Admixture 1	Admixture 2 (Percentage)	Retarder2 (gms)	Glowing Powder (gms)		
Reatarder 2 Mixes															
20	100	135	10	15	30	-	3	10	-	-	2	0.5	-		
21	100	135	10	15	30	-	3	10	-	-	2	1	-		
22	100	135	10	15	30	-	3	10	-	-	2	2	-		
23	100	135	10	15	30	-	3	10	-	-	2	3	-		
Glowing Mixes															
24	100	135	10	15	30	5	3	10	-	-	2	-	10		
25	100	135	10	15	30	5	3	10	-	-	2	-	15		
26	100	135	10	15	30	5	3	10	-	-	2	-	20		
27	100	135	10	15	30	5	3	10	-	-	2	-	25		
28	100	135	10	15	30	5	3	10	-	-	2	-	30		
Adjusting Crack Mixes															
29	100	135	10	15	25	5	3	10	0.5	-	2	-	-		
30	100	135	5	15	30	5	3	10	0.5	-	2	-	-		
31	100	135	10	15	40	5	3	10	0.5	-	2	-	-		
32	100	135	10	15	50	5	3	10	0.5	-	2	-	-		
Mixed of Admixtures															
33	100	135	10	15	30	5	3	10	0.5	1	1	-	-		
Fiber 1 Mixes															
34	100	135	10	15	30	5	-	10	0.5	-	2	-	-		
35	100	135	10	15	30	5	1.5	10	0.5	-	2	-	-		
36	100	135	10	15	30	5	3	10	0.5	-	2	-	-		
Fibers 2 Mixes															
37	100	135	10	15	30	5	1.5	10	0.5	-	2	-	-		
38	100	135	10	15	30	5	3	10	0.5	-	2	-	-		

**Notice:**

A = Silicate solution

B = Silica powder 1

C= Metal oxide

## **Matrix No. 1**

**Table 1**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	10
Fibers	3
H <sub>2</sub> O	10



**Fig. 3.1.1 shows the layout of sample mix no.1**

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 7-12 min.

### **Ease of Application**

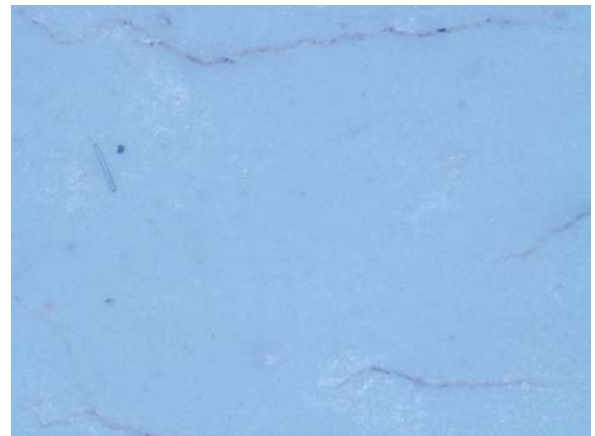
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface texture: medium roughness
- Score: 3.5



**Fig. 3.1.2 shows the small cracks developed**

### **Cracks development**

- Cracks developed parallel to the sample centerline. Medium to large sized cracks are spread all over the sample.
- Score: 3.5



**Fig. 3.1.3 digital microscopic picture showing the fine cracks (scale 25  $\mu$ m)**

## **Matrix No. 2**

**Table 2**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10



**Fig. 3.2.1 shows the layout of sample mix no. 2**

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 7-12 min.

### **Ease of Application**

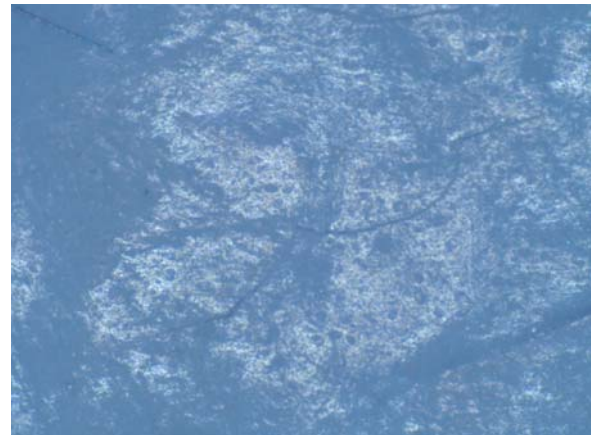
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: Medium to high roughness
- Score: 3.5



**Fig. 3.2.2 shows no cracks under visual observation**

### **Cracks development**

- Cracks developed diagonally to the sample centerline. The cracks are small sized cracks with slightly lower elevations than the surrounding surface.
- Score: 2.0



**Fig. 3.2.3 digital microscopic picture showing minimal cracks (scale 25  $\mu$ m)**

### **Matrix No. 3**

**Table 3**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
H <sub>2</sub> O	7.5

#### **Workability**

- Poor workability
- Medium brush marks
- Setting time: less than 7 min.

#### **-Ease of Application**

- Mix is medium heavy or clumsy
- Can be hardly applied using brush or roller
- Surface texture: low roughness
- Score: 1.0

#### **Cracks development**

- Interconnected cracks that form rectangular shapes developed all over the sample surface. The cracks are large sized cracks with highly lower elevations than the surrounding surface.
- Score: 5



**Fig. 3.3.1 shows the layout of sample mix no. 3**



**Fig. 3.3.2 shows large cracks under visual observation**



**Fig. 3.3.3 digital microscopic picture showing block cracks (scale 25 μm)**

## **Matrix No. 4**

**Table 4**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10



**Fig. 3.4.1 shows the layout of sample mix no. 4**

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 10-12 min.

### **-Ease of Application**

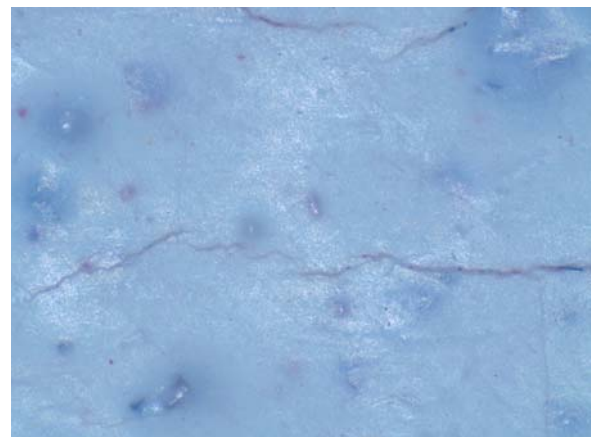
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: Medium roughness
- Score: 3.5



**Fig. 3.4.2 shows no cracks under visual observation**

### **Cracks development**

- Cracks developed parallel to the sample centerline. Medium sized cracks are spotted in different places of the sample.
- Score: 2.5



**Fig. 3.4.3 digital microscopic picture showing medium sized cracks (scale 25  $\mu$ m)**



## **Matrix No. 5**

**Table 5**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	15

### **Workability**

- High workability
- No brush marks
- Setting time: 12-15 min.

### **Ease of Application**

- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: medium to high roughness
- Score: 3.0

### **Cracks development**

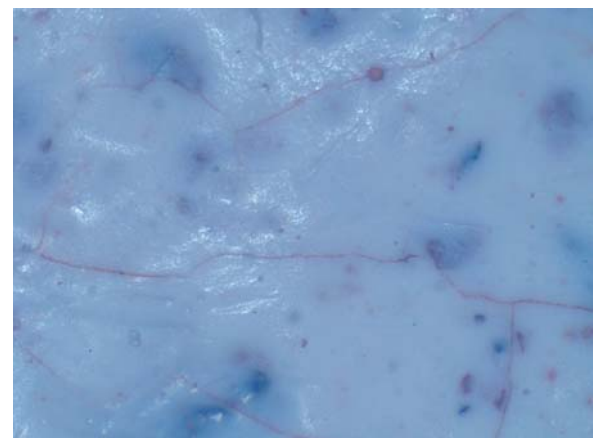
- Transverse and longitudinal cracks are developed in different parts of the sample. The cracks are small to medium sized cracks with lots of depressions all over the sample.
- Score: 2.5



**Fig. 3.5.1 shows the layout of sample mix no. 5**



**Fig. 3.5.2 shows no cracks under visual observation**



**Fig. 3.5.3 digital microscopic picture showing small sized longitudinal cracks (scale 25  $\mu$ m)**



## **Matrix No. 6**

**Table 6**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	20



**Fig. 3.6.1 shows the layout of sample mix no. 6**

### **Workability**

- Very high workability
- No brush marks
- Setting time: 15-20 min.

### **Ease of Application**

- Mix is very thing and form patches
- Can be easily applied using brush or roller
- Surface Texture: High roughness
- Score: 4.5



**Fig. 3.6.2 shows no cracks under visual observation**

### **Cracks development**

- Transverse and longitudinal cracks are developed in different parts of the sample. The cracks are very small sized cracks with lots of depressions all over the sample.
- Score: 2.0



**Fig. 3.6.3 digital microscopic picture showing minimal cracks (scale 25  $\mu$ m)**

## **Matrix No. 7**

**Table 7**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10
Retarder 1	0.25



**Fig. 3.7.1 shows mix no. 7 with two layers at the left side and one layer at the right side**

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 20-25 min.

### **Ease of Application**

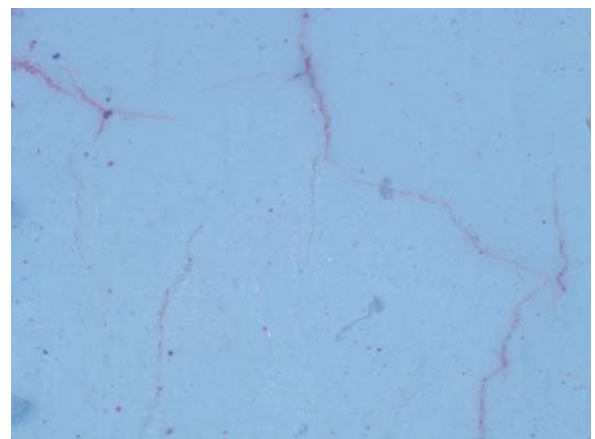
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 3.5

### **Cracks development**

- Transverse and diagonal cracks are developed all over the sample. The cracks are medium to large sized cracks that can be easily observed.
- Score: 4.0



**Fig. 3.7.2 shows fine longitudinal cracks all across the sample**



**Fig. 3.7.3 digital microscopic picture showing medium sized cracks (scale 25 μm)**

## **Matrix No. 8**

**Table 8**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10
Retarder 1	0.5



**Fig. 3.8.1 shows the layout of sample mix no. 8**

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 50- 60 min.

### **Ease of Application**

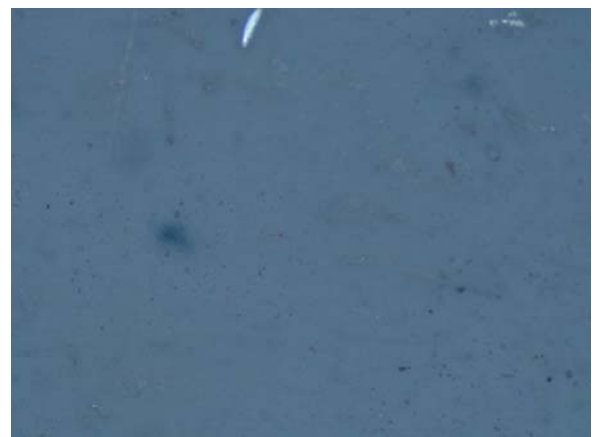
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 3.5



**Fig. 3.8.2 shows no cracks under visual observation**

### **Cracks development**

- No cracks are can be noticed. Very few depressions can be seen in few parts of the sample.
- Score: 0.5



**Fig. 3.8.3 digital microscopic picture showing no cracks (scale 25  $\mu$ m)**

## **Matrix No. 9**

**Table 9**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Alu. Fibers	3
H2O	10
Retarder 1	1



**Fig. 3.9.1 shows the layout of sample mix no. 9**

### **Workability**

- High workability
- No brush marks
- Setting time: 60-75 min.

### **Ease of Application**

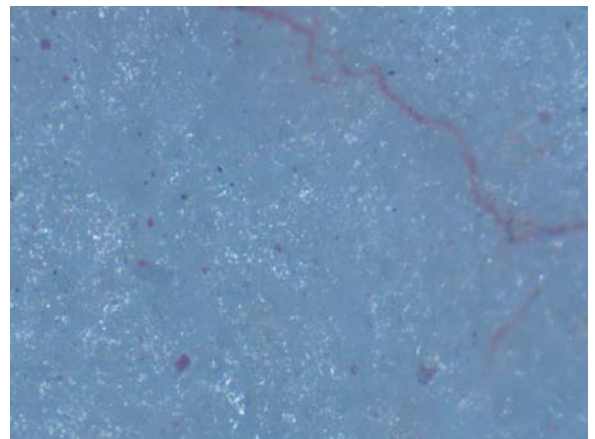
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 3.5



**Fig. 3.9.2 shows almost no cracks under visual observation**

### **Cracks development**

- Longitudinal and diagonal cracks are developed all over the sample. The cracks are medium to large sized cracks that can be easily observed.
- Score: 4.0



**Fig. 3.9.3 digital microscopic picture showing minimal cracks (scale 25  $\mu$ m)**

## **Matrix No. 10**

**Table 10**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10
Retarder 1	2



**Fig. 3.10.1 shows layout of sample mix no. 10**

### **Workability**

- High workability
- No brush marks
- Setting time: 75- 95 min.

### **Ease of Application**

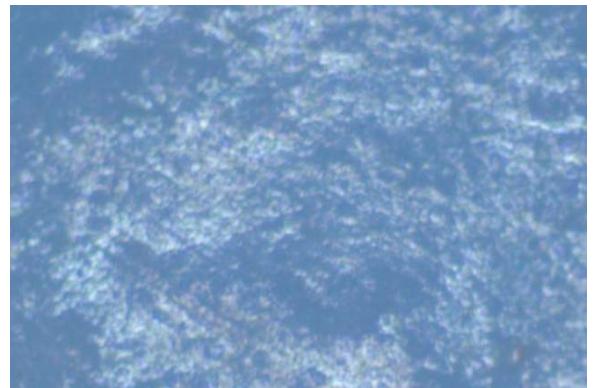
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: high roughness
- Score: 4.0

### **Cracks development**

- Small, bowl-shaped depressions in the sample surface are developed on the sample surface. Lots of holes can be observed on the sample surface along with very few cracks.
- Score: 3.5



**Fig. 3.10.2 shows depressions and holes in the surface**



**Fig. 3.10.3 digital microscopic picture showing no cracks (scale 25  $\mu$ m)**

## **Matrix No. 11**

**Table 11**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10
Retarder 1	4

### **Workability**

- High to very high workability
- No brush marks
- Setting time: 100- 115 min

### **Ease of Application**

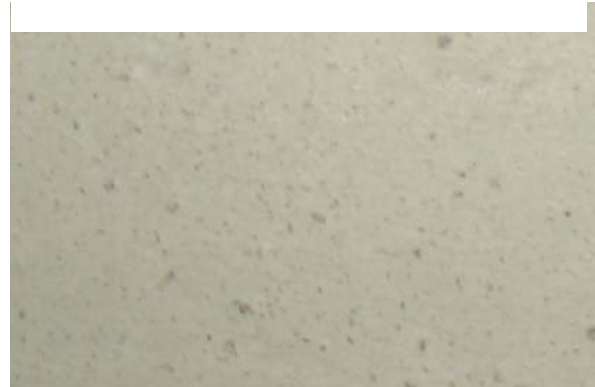
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: high roughness
- Score: 4.0

### **Cracks development**

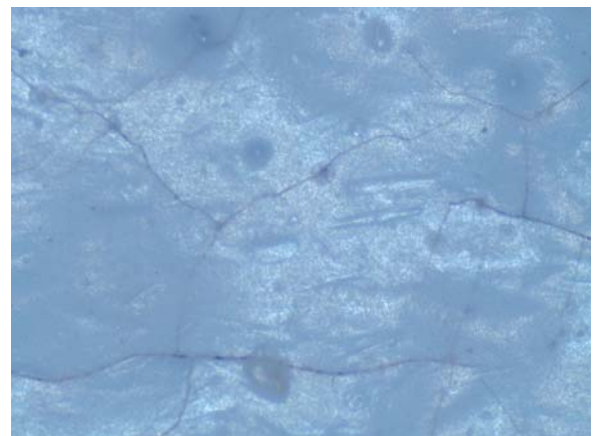
- Many depressions and holes observed on the sample surface. Interconnected cracks of small to medium size have been developed on different spots of the sample.
- Score: 3.5



**Fig. 3.11.1 shows layout of sample mix no. 11**



**Fig. 3.11.2 shows lots of small depressions and holes**



**Fig. 3.11.3 digital microscopic picture showing no cracks (scale 25  $\mu$ m)**



## **Matrix No. 12**

**Table 12**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 1	3.1

### **Workability**

- High workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 3.5

### **Cracks development**

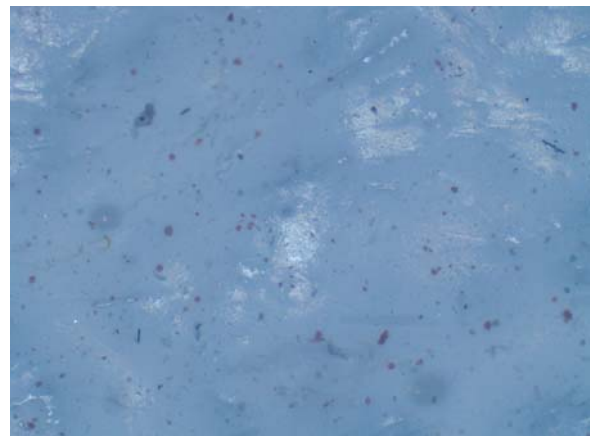
- No cracks can be observed on the sample surface. Few depressions are developed in various parts of the sample.
- Score: 0.5



**Fig. 3.12.1 shows layout of sample mix no. 12**



**Fig. 3.12.2 shows no cracks under visual observation**



**Fig. 3.12.3 digital microscopic picture showing no cracks (scale 25  $\mu$ m)**

## **Matrix No. 13**

**Table 13**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 1	6.2

### **Workability**

- High workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 4.0

### **Cracks development**

- Very fine transverse cracks can be seen under the microscope with almost no depressions.
- Score: 0.5



**Fig. 3.13.1 shows layout of sample mix no. 13**



**Fig. 3.13.2 shows no cracks under visual observation**



**Fig. 3.13.3 digital microscopic picture showing very fine cracks < 1  $\mu$ m (scale 25  $\mu$ m)**



## **Matrix No. 14**

**Table 14**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 1	9.3

### **Workability**

- High workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: medium roughness
- Score: 4.5

### **Cracks development**

- Interconnected cracks are developed all over the sample surface. Longitudinal cracks are large sized cracks that can be clearly seen on Fig 3.14.2.
- Score: 4.0



**Fig. 3.14.1 shows layout of sample mix no. 14**



**Fig. 3.14.2 shows the major cracks in the sample surface**



**Fig. 3.14.3 digital microscopic picture showing block cracks (scale 25  $\mu$ m)**

## **Matrix No. 15**

**Table 15**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	12.4

### **Workability**

- Very high workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

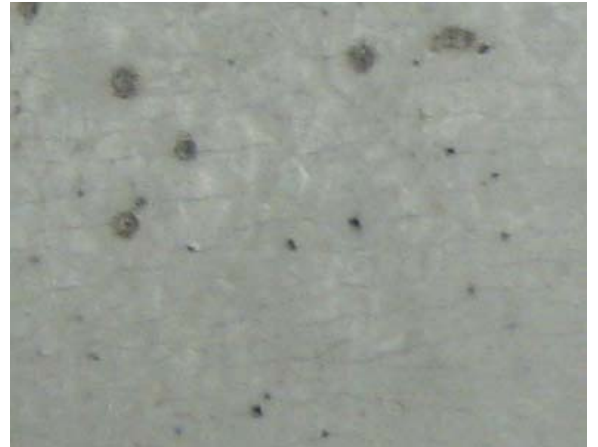
- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: very high roughness
- Score: 4.5

### **Cracks development**

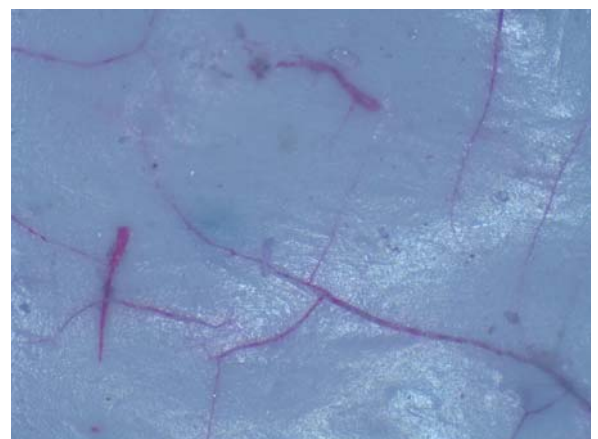
- Large interconnected cracks along with large holes are developed all over the sample surface. This is can be illustrated on Fig. 3.15.2
- Score: 5.0



**Fig. 3.15.1** shows layout of sample mix no. 15



**Fig. 3.15.2** shows large cracks and large pot holes all across the sample surface



**Fig. 3.15.3** digital microscopic picture showing large cracks (scale 25 μm)

## **Matrix No. 16**

**Table 16**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	3.1

### **Workability**

- High workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 4.0

### **Cracks development**

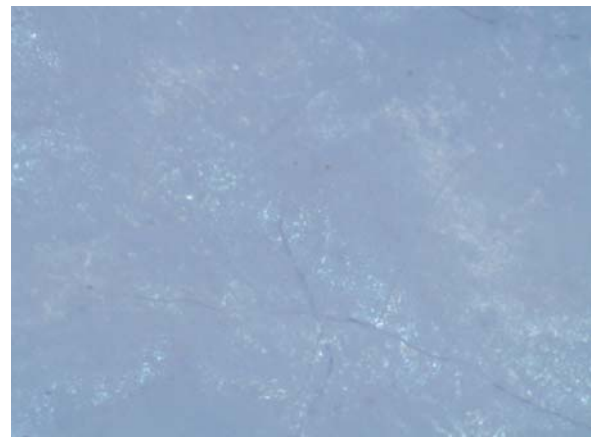
- Very fine cracks can be spotted on the sample surface. Few depressions are developed in various parts of the sample.
- Score: 1.0



**Fig. 3.16.1 shows layout of sample mix no. 16**



**Fig. 3.16.2 shows no cracks under visual observation**



**Fig. 3.16.3 digital microscopic picture showing minimal depressions (scale 25  $\mu$ m)**

## **Matrix No. 17**

**Table 17**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	6.2

### **Workability**

- High workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 4.0

### **Cracks development**

- Almost no cracks and very few depressions can be seen on the sample surface.
- Score: 0.5



**Fig. 3.17.1 shows layout of sample mix no. 17**



**Fig. 3.17.2 shows no cracks under visual observation**



**Fig. 3.17.3 digital microscopic picture showing no cracks (scale 25 μm)**

## **Matrix No. 18**

**Table 18**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	9.3

### **Workability**

- High workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: medium roughness
- Score: 4.5

### **Cracks development**

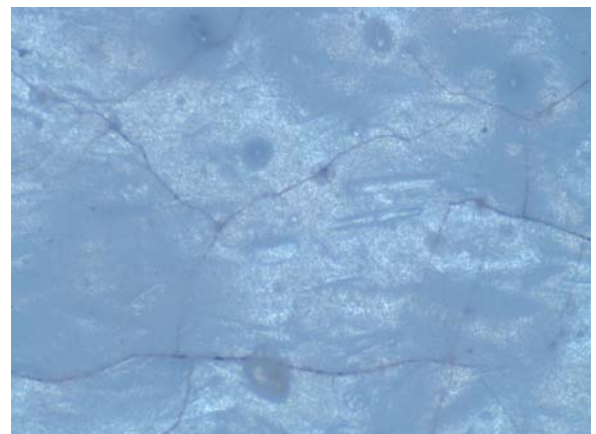
- Interconnected cracks along with depressions are developed all over the sample surface. The cracks are large sized cracks that can be identified by visual observation.
- Score: 4.5



**Fig. 3.18.1 shows layout of sample mix no. 18**



**Fig. 3.18.2 shows cracks all across the sample surface**



**Fig. 3.18.3 digital microscopic picture showing cracks and depressions (scale 25  $\mu$ m)**

## **Matrix No. 19**

**Table 19**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	12.4

### **Workability**

- Very high workability
- No brush marks
- Setting time: 50- 60 min

### **Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: very high roughness
- Score: 4.5

### **Cracks development**

- Large interconnected cracks along with few depressions are developed all over the sample surface. This can be illustrated through Fig.3.19.2
- Score: 5.0



**Fig. 3.19.1 shows layout of sample mix no. 19**



**Fig. 3.19.2 shows large cracks all across the sample surface**



**Fig. 3.19.3 digital microscopic picture showing large block cracks (scale 25 μm)**



## **Matrix No. 20**

**Table 20**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10
Admixture 2	6.1
Retarder 2	0.5

### **Workability**

- High workability
- No brush marks
- Setting time: 55- 65 min

### **Ease of Application**

- Mix is inconsistent and have some lumps
- Can be easily applied using brush or roller
- Surface Texture: high roughness and glossy
- Score: 4.0

### **Cracks development**

- Almost no cracks developed on the sample surface. Lots of depressions can be seen due to the inconsistency of the mix.
- Score: 1.0



**Fig. 3.20.1 shows layout of sample mix no. 20**



**Fig. 3.20.2 shows almost no cracks under visual observation**



**Fig. 3.20.3 digital microscopic picture showing minimal depressions (scale 25 µm)**

## **Matrix No. 21**

**Table 21**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10
Admixture 2	6.1
Retarder 2	1

### **Workability**

- High workability
- No brush marks
- Setting time: 75- 80 min

### **Ease of Application**

- Mix is inconsistent and full of lumps
- Can be easily applied using brush or roller
- Surface Texture: high roughness and glossy
- Score: 4.5

### **Cracks development**

- Large depressions can be spotted all over the sample surface with very fine and few cracks as shown in Fig. 3.12.2
- Score: 1.5



**Fig. 3.21.1 shows layout of sample mix no. 21**



**Fig. 3.21.2 shows almost pot holes and depressions all over the sample surface**



**Fig. 3.21.3 digital microscopic picture showing almost no cracks with minimal depressions (scale 25  $\mu$ m)**



## **Matrix No. 22**

**Table 22**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H <sub>2</sub> O	10
Admixture 2	6.1
Retarder 2	2

### **Workability**

- Very high workability
- No brush marks
- Setting time: 90- 100 min

### **Ease of Application**

- Mix is lumpy and inconsistent
- Can be easily applied using brush or roller
- Surface Texture: high roughness and glossy
- Score: 4.5

### **Cracks development**

- Large depressions can be seen all over the sample surface with very fine cracks
- Score: 1.5



**Fig. 3.22.1 shows layout of sample mix no. 22**



**Fig. 3.22.2 shows almost no cracks with lots of depressions and holes**



**Fig. 3.22.3 digital microscopic picture showing very fine cracks < 1  $\mu$ m (scale 25  $\mu$ m)**

## **Matrix No. 23**

**Table 23**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
Fibers	3
H2O	10
Admixture 2	6.1
Retarder 2	3

### **Workability**

- Very high workability
- No brush marks
- Setting time: 110-120 min

### **Ease of Application**

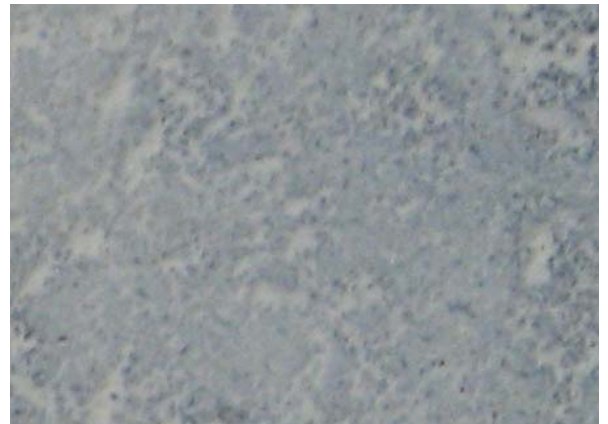
- Mix is very lumpy and inconsistent
- Can be easily applied using brush or roller
- Surface Texture: high roughness and glossy
- Score: 4.5

### **Cracks development**

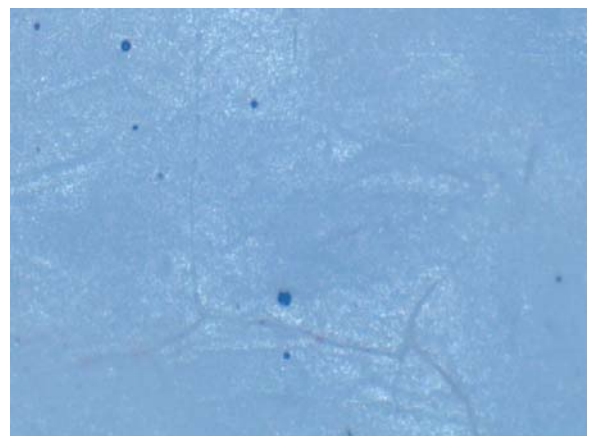
- Large depressions can be seen all over the sample surface due to the inconsistency of the mix. Also, few cracks have been spotted.
- Score: 2.5



**Fig. 3.23.1 shows layout of sample mix no. 23**



**Fig. 3.23.2 shows almost no cracks and lots of surface depressions**



**Fig. 3.23.3 digital microscopic picture showing medium sized cracks (scale 25  $\mu$ m)**

## **Matrix No. 24**

**Table 24**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Admixture 2	2%
Glowing Powder	10

### **Workability**

- High workability
- No brush marks
- Setting time: 10-15 min.

### **Ease of Application**

- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: Medium roughness
- Score: 4.0

### **Cracks development**

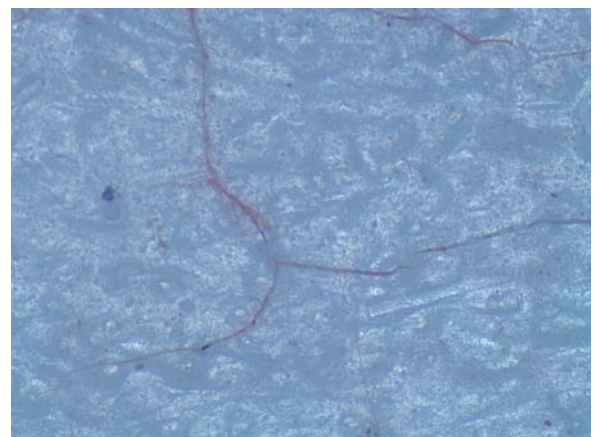
- Cracks developed parallel and perpendicular to the sample centerline. Medium sized cracks along with depressions are spread all over the sample surface.
- Score: 3.5



**Fig. 3.24.1 shows layout of sample mix no. 24**



**Fig. 3.24.2 shows the sample in the dark with very little glowing properties**



**Fig. 3.24.3 digital microscopic picture showing longitudinal and transverse cracks (scale 25  $\mu$ m)**

## **Matrix No. 25**

**Table 25**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Admixture 2	2%
Glowing Powder	15

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 7-12 min.

### **Ease of Application**

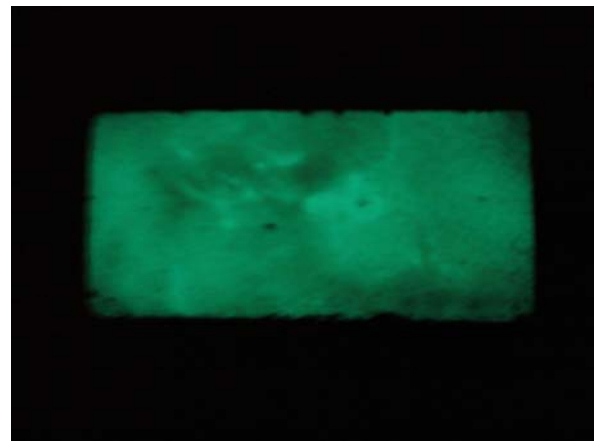
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: Medium to high roughness
- Score: 3.5

### **Cracks development**

- Diagonal Cracks developed all over the sample surface. Medium sized cracks along with very few depressions can be clearly seen on Fig 3.25.3
- Score: 3.5



**Fig. 3.25.1 shows layout of sample mix no. 25**



**Fig. 3.25.2 shows the sample in the dark with high glowing properties**



**Fig. 3.25.3 digital microscopic picture showing diagonal cracks (scale 25 µm)**

## **Matrix No. 26**

**Table 26**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Admixture2	2%
Glowing Powder	20

### **Workability**

- Medium workability
- No brush marks
- Setting time: 5-10 min.

### **Ease of Application**

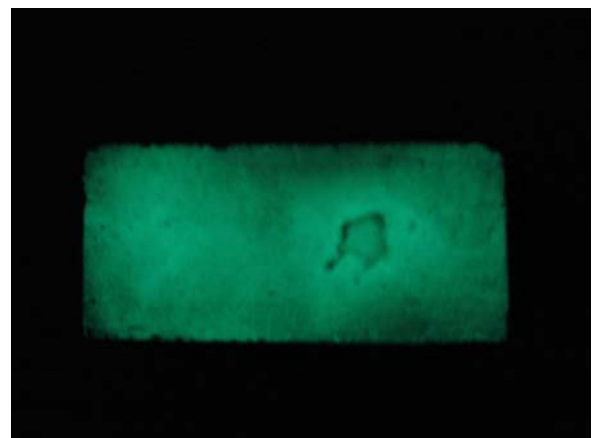
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: High roughness
- Score: 3.0

### **Cracks development**

- Few cracks developed along with few depressions which can be illustrated through Fig 3.26.3
- Score: 1.5



**Fig. 3.26.1 shows layout of sample mix no. 26**



**Fig. 3.26.2 shows the sample in the dark with high glowing properties**



**Fig. 3.26.3 digital microscopic picture showing fine cracks on the sample surface (scale 25 μm)**



## **Matrix No. 27**

**Table 27**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Admixture 2	2%
Glowing Powder	25

### **Workability**

- Medium workability
- No brush marks
- Setting time: 5-10 min.

### **Ease of Application**

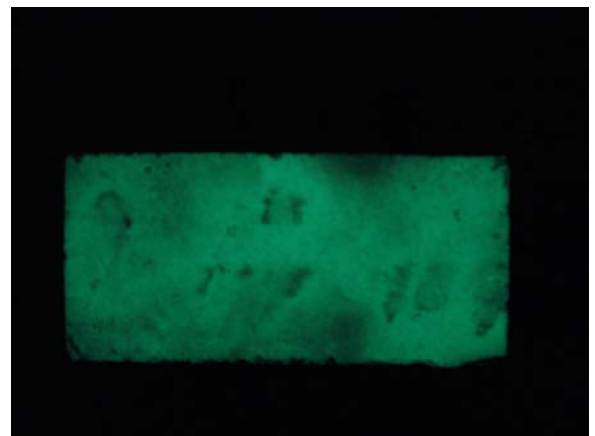
- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: High roughness
- Score: 3.0

### **Cracks development**

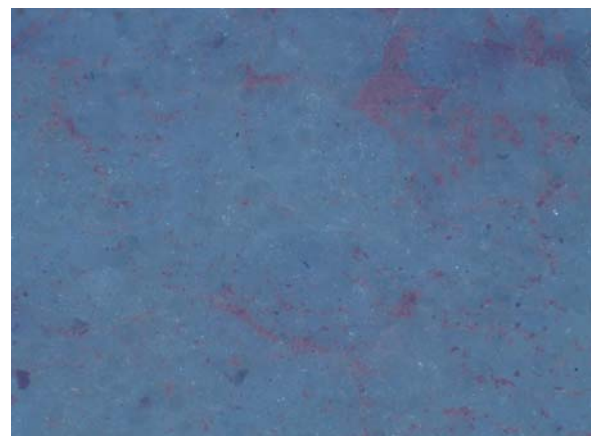
- Few cracks along with corrugation developed across the sample surface which can be illustrated through Fig 3.27.3
- Score: 3.5



**Fig. 3.27.1 shows layout of sample mix no. 27**



**Fig. 3.27.2 shows the sample with high glowing properties in the dark**



**Fig. 3.27.3 digital microscopic picture showing few cracks along with heavy depressions (scale 25 μm)**

**Matrix No. 28****Table 28**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Admixture 2	2%
Glowing Powder	30

**Workability**

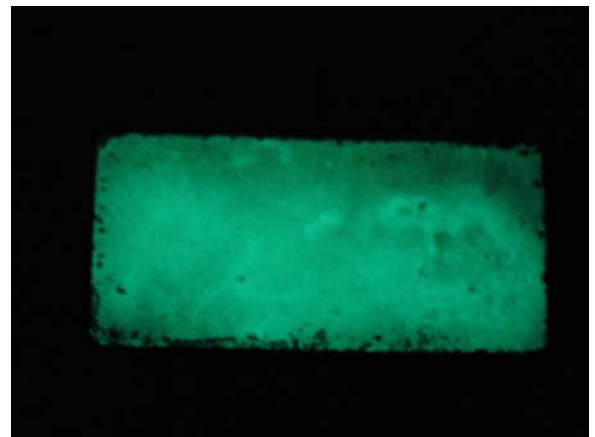
- Medium workability
- No brush marks
- Setting time: 5-10 min.

**Ease of Application**

- Can be easily mixed
- Can be easily applied using brush or roller
- Surface Texture: High to very high roughness
- Score: 3.0

**Cracks development**

- Medium sized transverse cracks developed on different parts of the sample surface. Also, few depressions can be spotted in random places of the sample
- Score: 3.0

**Fig. 3.28.1 shows layout of sample mix no. 28****Fig. 3.28.2 shows the sample in the dark with high glowing properties****Fig. 3.28.3 digital microscopic picture showing longitudinal cracks (scale 25 μm)**

## **Matrix No. 29**

**Table 29**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	25
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

### **Workability**

- High workability
- No brush marks
- Setting time: 10-13 min.

### **Ease of Application**

- Mix is thin and of medium consistency
- Can be easily applied using brush or roller
- Surface Texture: High roughness
- Score: 3.5

### **Cracks development**

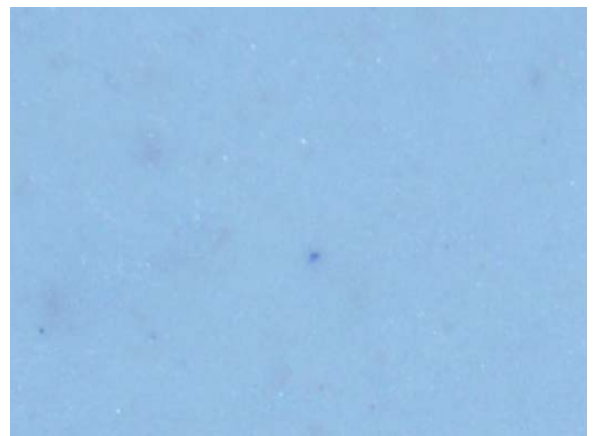
- No cracks are can be spotted on the sample surface. Some depressions are developed in various parts of the sample.
- Score: 1.0



**Fig. 3.29.1 shows layout of sample mix no. 29**



**Fig. 3.29.2 shows almost no cracks under visual observation**



**Fig. 3.29.3 digital microscopic picture showing no cracks (scale 25 μm)**



## **Matrix No. 30**

**Table 30**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	30
Ground Filler	25
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 7-12 min.

### **Ease of Application**

- Mix is of medium consistency with some lumps
- Can be easily applied using brush or roller
- Surface Texture: High to very high roughness
- Score: 3.0

### **Cracks development**

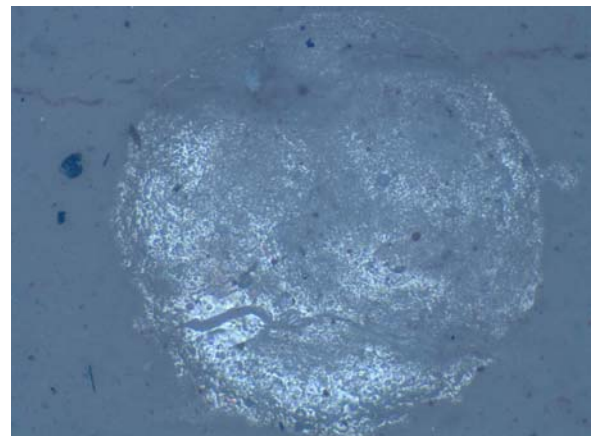
- Small size cracks are can be spotted on different parts of the sample surface. Lots of depressions are developed in various parts of the sample.
- Score: 2.5



**Fig. 3.30.1 shows layout of sample mix no. 30**



**Fig. 3.30.2 shows small cracks and depressions all over the sample surface**



**Fig. 3.30.3 digital microscopic picture showing a very big depression and minimal cracking (scale 25 μm)**

## **Matrix No. 31**

**Table 31**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	40
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

### **Workability**

- Low to Medium workability
- No brush marks
- Setting time: 5-10 min.

### **Ease of Application**

- Medium consistency with few lumps
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 2.5

### **Cracks development**

- Transverse and diagonal cracks are developed all over the sample. The cracks are medium to large sized cracks that can be spotted all over the sample under visual observation.
- Score: 4.0



**Fig. 3.31.1 shows layout of sample mix no. 31**



**Fig. 3.31.2 shows cracks all over the sample surface**



**Fig. 3.31.3 digital microscopic picture showing transverse cracks (scale 25 μm)**

## **Matrix No. 32**

**Table 32**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	50
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

### **Workability**

- Low to medium workability
- No brush marks
- Setting time: 5-8 min.

### **Ease of Application**

- Bad consistency with lots of lumps
- Can be applied using a roller as lumps stick to brush
- Surface Texture: Very high roughness
- Score: 2.0

### **Cracks development**

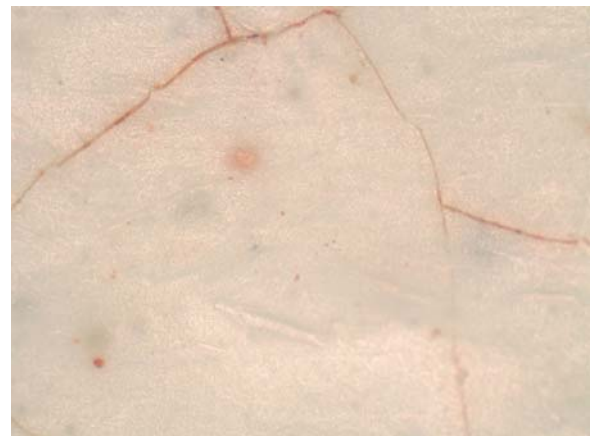
- Interconnected cracks are developed in different places of the sample. Large depressions can be spotted as shown in Fig. 3.32.2
- Score: 4.0



**Fig. 3.32.1 shows layout of sample mix no. 32**



**Fig. 3.32.2 shows heavy depressions all over the sample surface**



**Fig. 3.32.3 digital microscopic picture showing cracks all over the surface (scale 25 µm)**

**Matrix No. 33****Table 33**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	25
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 1	1%
Admixture 2	1%

**Workability**

- High workability
- No brush marks
- Setting time: 50- 60 min

**Ease of Application**

- Mix is very consistent and has smooth texture
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 4.0

**Cracks development**

- No cracks are can be spotted on the sample surface. Some depressions and holes are developed in various parts of the sample.
- Score: 2.0

**Fig. 3.33.1 shows layout of sample mix no. 33****Fig. 3.33.2 shows no cracks with few depressions on the sample surface****Fig. 3.33.3 digital microscopic picture showing no cracks (scale 25 µm)**

## **Matrix No. 34**

**Table 34**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	25
TiO <sub>2</sub>	5
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

### **Workability**

- High workability
- No brush marks
- Setting time: 50-60 min.

### **Ease of Application**

- Mix is consistent and has a smooth texture
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 4.0

### **Cracks development**

- No cracks can be seen on the sample surface. Very Few depressions are developed in various parts of the sample.
- Score: 0.5



**Fig. 3.34.1 shows layout of sample mix no. 34**



**Fig. 3.34.2 shows no cracks and smooth surface**



**Fig. 3.34.3 microscopic picture showing no cracks with very few depressions (scale 25 µm)**

## **Matrix No. 35**

**Table 35**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	25
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 50-60 min.

### **Ease of Application**

- Mix is very consistent and has a smooth texture
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 3.5

### **Cracks development**

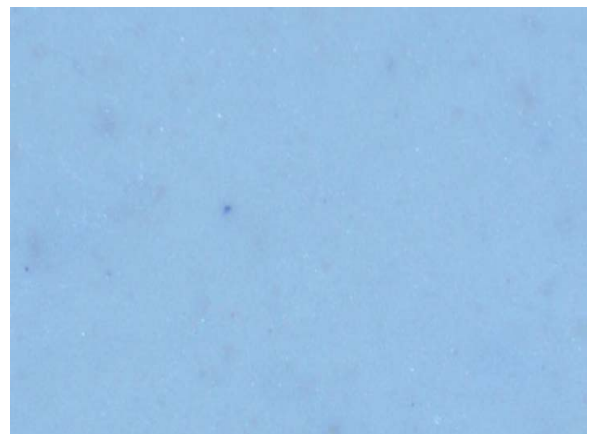
- Very few cracks less than 1  $\mu\text{m}$  are developed on the sample surface. Few depressions can be identified on different parts of the sample.
- Score: 1.0



**Fig. 3.35.1 shows layout of sample mix no. 35**



**Fig. 3.35.2 shows no cracks with few depressions**



**Fig. 3.35.3 digital microscopic picture showing no cracks (scale 25  $\mu\text{m}$ )**



### **Matrix No. 36**

**Table 36**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	25
TiO <sub>2</sub>	5
Fibers	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

#### **Workability**

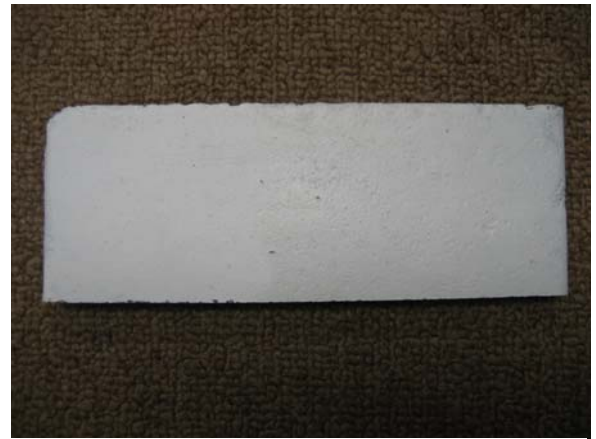
- Medium to high workability
- No brush marks
- Setting time: 50-60 min.

#### **Ease of Application**

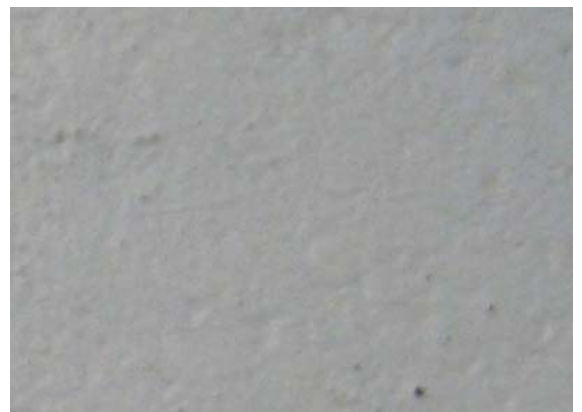
- Mix is consistent and has a smooth texture
- Can be easily applied using brush or roller
- Surface Texture: low roughness
- Score: 3.5

#### **Cracks development**

- Very few cracks less than 1  $\mu\text{m}$  are developed on the sample surface. Few large depressions can be seen in very few spots.
- Score: 1.0



**Fig. 3.36.1 shows layout of sample mix no. 36**



**Fig. 3.36.2 shows small cracks and depressions all over the sample surface**



**Fig. 3.36.3 digital microscopic picture showing no cracks (scale 25  $\mu\text{m}$ )**

## **Matrix No. 37**

**Table 37**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	25
TiO <sub>2</sub>	5
Fibers 2	1.5
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 50-60 min.

### **Ease of Application**

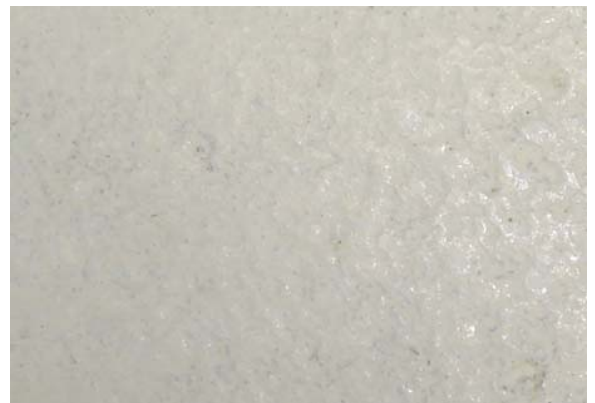
- Mix is very consistent and has a smooth texture
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 3.5

### **Cracks development**

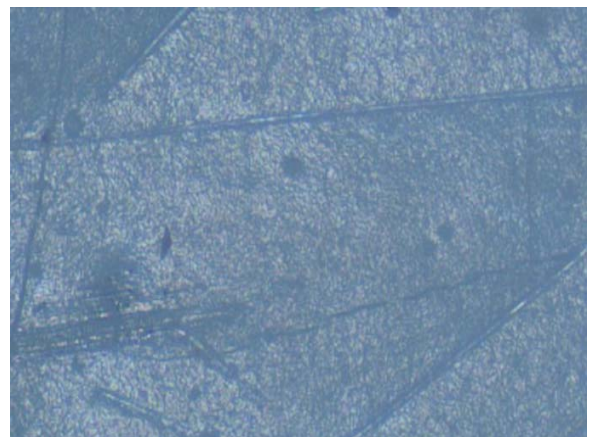
- Few cracks are developed on the sample surface. Large depressions were created on the fibers locations which can be clearly seen on Fig. 3.37.3
- Score: 2.0



**Fig. 3.37.1 shows layout of sample mix no. 37**



**Fig. 3.37.2 shows no cracks with few depressions**



**Fig. 3.37.3 microscopic picture showing depressions where the fibers are located (scale 25 μm)**



**Matrix No. 38****Table 38**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	135
Part C	10
Micro fiber	15
Ground Filler	25
TiO <sub>2</sub>	5
Fibers 2	3
H <sub>2</sub> O	10
Retarder 1	0.5
Admixture 2	2%

**Workability**

- Medium to high workability
- No brush marks
- Setting time: 50-60 min.

**Ease of Application**

- Mix is consistent and has a smooth texture
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 3.5

**Cracks development**

- Lots of large depressions were developed on the fibers locations which can be clearly seen on Fig. 3.38.3. Fine cracks can be seen on few parts of the sample.
- Score: 2.5

**Fig. 3.38.1 shows layout of sample mix no. 38****Fig. 3.38.2 shows no cracks with heavy depressions all over the sample surface****Fig. 3.38.3 digital microscopic picture showing cracks and heavy depressions (scale 25 μm)**

**Table 2.2 ( Silica Powder 2)**

Mix No.	Ingredients										Remarks
	A (gms)	B (gms)	C (gms)	Microfibers (gms)	Ground Filler (gms)	TiO <sub>2</sub> (gms)	Fibers1 (gms)	Fibers2 (gms)	H <sub>2</sub> O (gms)	Admixture 2	
<b><u>Base Mixes</u></b>											
39	100	120	10	15	30	-	1	-	10	-	
40	100	100	10	15	30	-	1	-	10	-	
<b><u>Titanium dioxide Mixes</u></b>											
41	100	120	10	15	30	5	1	-	10	-	Coarse TiO <sub>2</sub>
42	100	100	10	15	30	5	1	-	10	-	Coarse TiO <sub>2</sub>
43	100	100	10	15	30	10	1	-	10	-	Coarse TiO <sub>2</sub>
<b><u>Admixture 2 Mixes</u></b>											
44	100	100	10	15	30	5	1	-	10	1%	
45	100	100	10	15	30	5	1	-	10	2%	
46	100	100	10	15	30	5	1	-	10	3%	
47	100	100	10	15	30	5	1	-	4	3.3 gms	
48	100	120	10	15	30	5	1	-	4	3.3 gms	
<b><u>Fibers 2 Mixes</u></b>											
49	100	120	10	15	30	5	1	1.5	4	3.3 gms	
50	100	120	10	15	30	5	1	3	4	3.3 gms	

**Notes:**

A = Silicate Solution

B = Silicate Powder 2

D= Metal oxide

## **Matrix No. 39**

**Table 39**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	120
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	-
Fibers	1
H <sub>2</sub> O	10



**Fig. 3.39.1** shows layout of sample mix no. 39

### **Workability**

- Very low workability
- Heavy brush marks
- Setting time: less than 7 min.

### **Ease of Application**

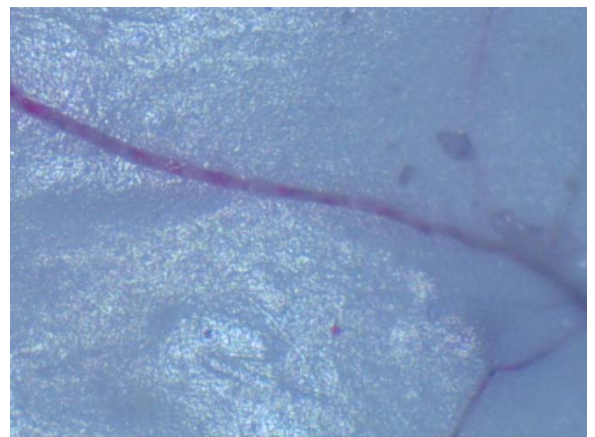
- Mix is like paste with no lumps
- Can be hardly applied using brush or roller
- Surface Texture: Low to Medium roughness
- Score: 1.5



**Fig. 3.39.2** shows large cracks all over the sample surface

### **Cracks development**

- Large interconnected and diagonal cracks along with some depressions are developed all over the sample surface.
- Score: 5.0



**Fig. 3.39.3** digital microscopic picture showing a big transverse crack (scale 25  $\mu$ m)

**Matrix No. 40****Table 40**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	100
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	-
Fibers	1
H <sub>2</sub> O	10

**Workability**

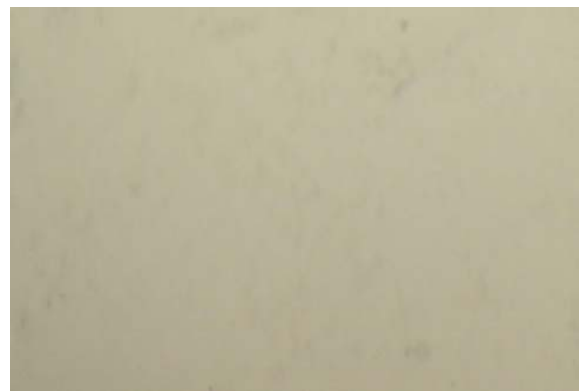
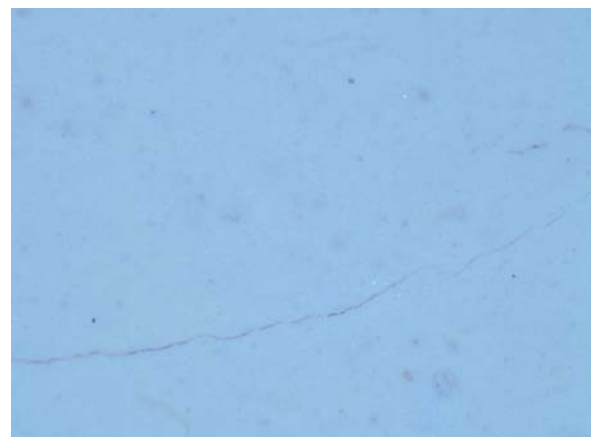
- Medium workability
- No brush marks
- Setting time: 7-12 min.

**Ease of Application**

- Mix is of consistent with no lumps
- Can be easily applied using brush or roller
- Surface Texture: low to medium roughness
- Score: 3.0

**Cracks development**

- Small to medium size cracks diagonally developed all over the sample surface. Also, few depressions were developed across the surface.
- Score: 3.5

**Fig. 3.40.1 shows layout of sample mix no. 40****Fig. 3.40.2 shows small cracks and depressions all over the sample surface****Fig. 3.40.3 digital microscopic picture showing medium size transverse cracks (scale 25  $\mu$ m)**

## **Matrix No. 41**

**Table 41**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	120
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
H <sub>2</sub> O	10



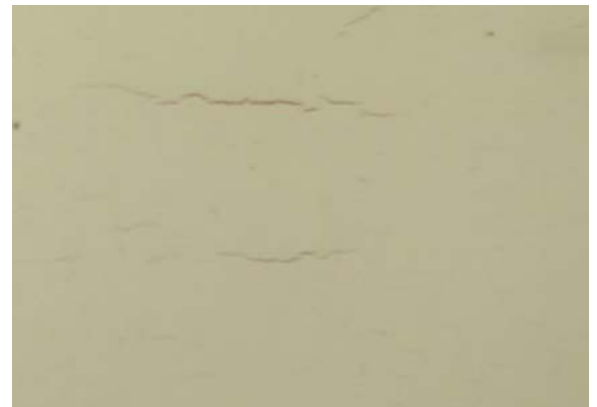
**Fig. 3.41.1 shows layout of sample mix no. 41**

### **Workability**

- Very low workability
- Heavy brush marks
- Setting time: less than 7 min.

### **Ease of Application**

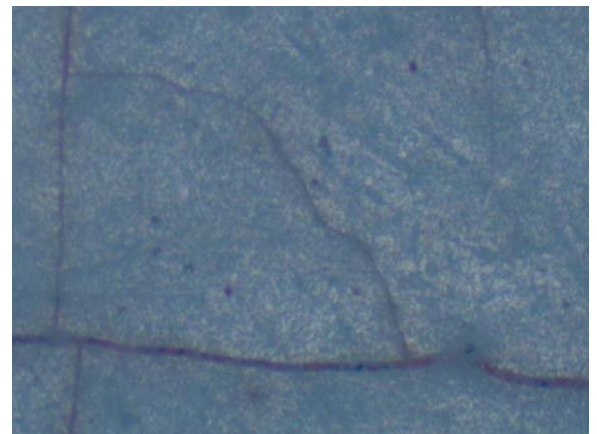
- Mix is of medium consistency with no lumps
- Can be hardly applied using brush or roller
- Surface Texture: medium roughness
- Score: 1.0



**Fig. 3.41.2 shows very big cracks along with medium size cracks all over the sample surface**

### **Cracks development**

- Large interconnected cracks forming rectangular shapes along with some depressions are developed all over the sample surface.
- Score: 5.0



**Fig. 3.41.3 digital microscopic picture showing large longitudinal and transverse cracks (scale 25  $\mu$ m)**

**Matrix No. 42****Table 42**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	100
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
H <sub>2</sub> O	10

**Fig. 3.42.1 shows layout of sample mix no. 42****Workability**

- Very low to low workability
- Heavy brush marks
- Setting time: less than 7 min.

**Ease of Application**

- Mix is of medium consistency with no lumps
- Can be hardly applied using brush or roller
- Surface Texture: low to medium roughness
- Score: 1.0

**Fig. 3.42.2 shows almost no cracks under the visual observation****Cracks development**

- Small size cracks can be spotted on different parts of the sample surface. Lots of depressions are developed in various parts of the sample.
- Score: 2.5

**Fig. 3.42.3 digital microscopic picture showing no cracks with depressions (scale 25 µm)**



**Matrix No. 43****Table 43**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	100
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	10
Fibers	1
H <sub>2</sub> O	10

**Workability**

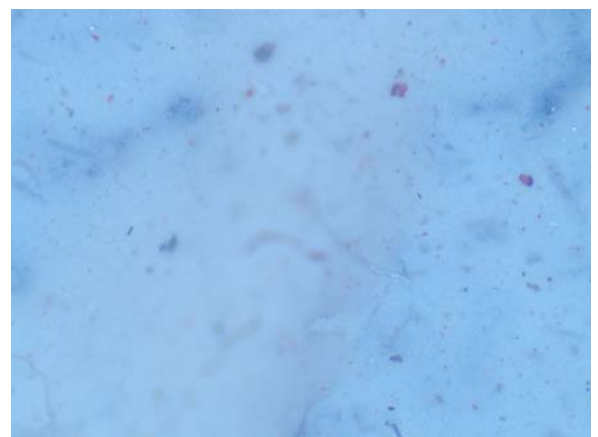
- Very low workability
- Heavy brush marks
- Setting time: less than 7 min.

**Ease of Application**

- Mix is of medium to high consistency with no lumps
- Can be hardly applied using brush or roller
- Surface Texture: High roughness
- Score: 1.5

**Cracks development**

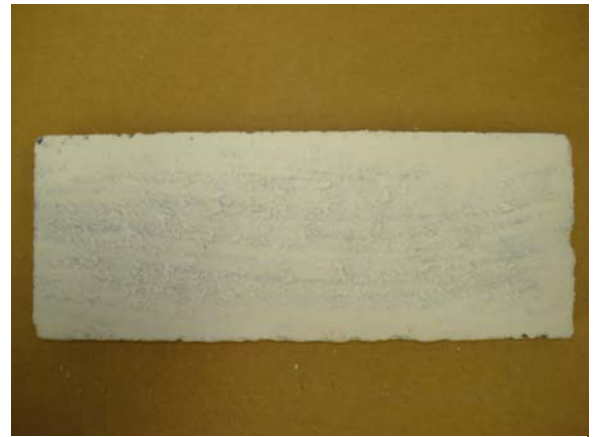
- Medium to large size cracks can be observed on various parts of the sample surface. Lots of depressions are developed all over the sample
- Score: 4.0

**Fig. 3.43.1 shows layout of sample mix no. 43****Fig. 3.43.2 shows medium size cracks all over the sample surface****Fig. 3.43.3 digital microscopic picture different light shades due to surface roughness (scale 25 µm)**

## **Matrix No. 44**

**Table 44**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	100
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
H <sub>2</sub> O	10
Admixture 2	1%



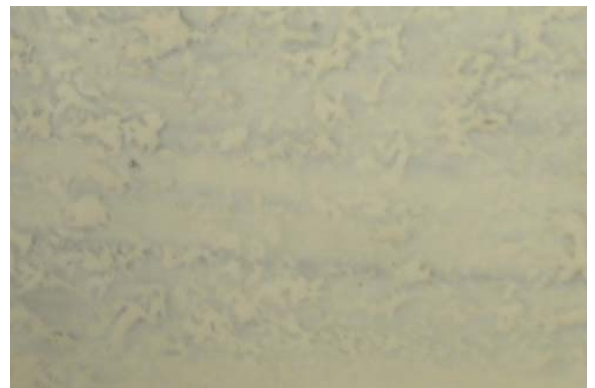
**Fig. 3.44.1 shows layout of sample mix no. 44**

### **Workability**

- Very high workability
- No brush marks
- Setting time: 60-70 min.

### **Ease of Application**

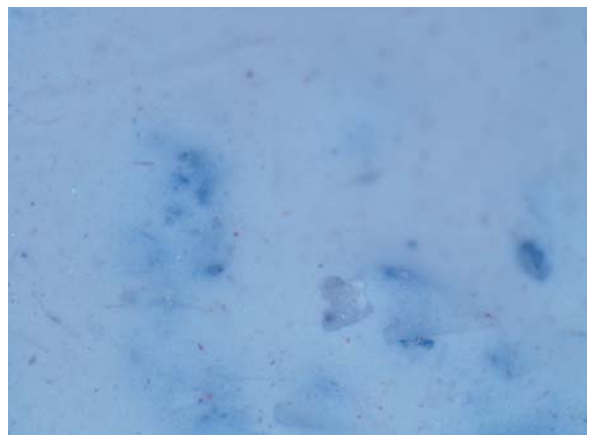
- Mix is of very thin and has a very low viscosity
- Can be easily applied using brush or roller
- Surface Texture: Medium roughness
- Score: 4.5



**Fig. 3.44.2 shows no cracks and depressions all over the sample surface**

### **Cracks development**

- Almost no cracks developed on the sample surface. Corrugation and surface shoving can be seen due to the thin mix.
- Score: 1.0



**Fig. 3.44.3 digital microscopic picture showing no cracks (scale 25 µm)**



## **Matrix No. 45**

**Table 45**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	100
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
H <sub>2</sub> O	10
Admixture 2	2%



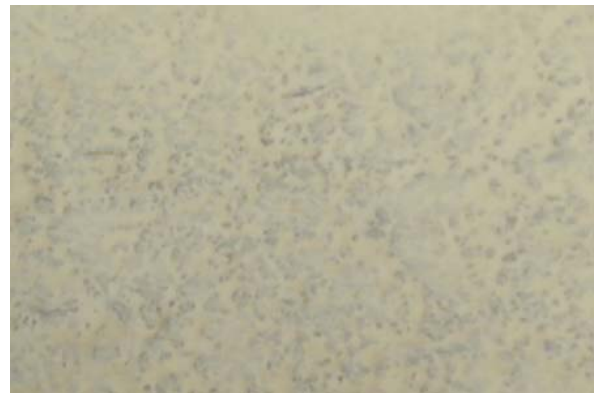
**Fig. 3.45.1 shows layout of sample mix no. 45**

### **Workability**

- Very high workability
- No brush marks
- Setting time: 80-90 min.

### **Ease of Application**

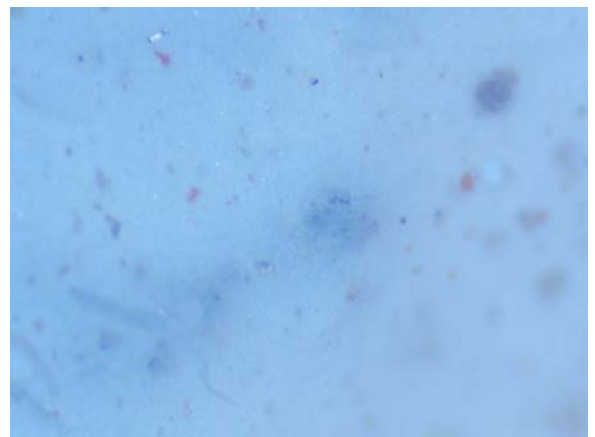
- Mix is very thin and inconsistent
- Can be easily applied using brush or roller
- Surface Texture: Medium roughness
- Score: 5.0



**Fig. 3.45.2 shows no cracks depressions all over the sample surface**

### **Cracks development**

- Almost no cracks developed on the sample surface. Heavy surface depression can be seen due to the thin mix.
- Score: 1.0



**Fig. 3.45.3 digital microscopic picture showing no cracks (scale 25 μm)**

**Matrix No. 46****Table 46**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	100
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
H <sub>2</sub> O	10
Admixture 2	3%

**Fig. 3.46.1** shows layout of sample mix no. 46**Workability**

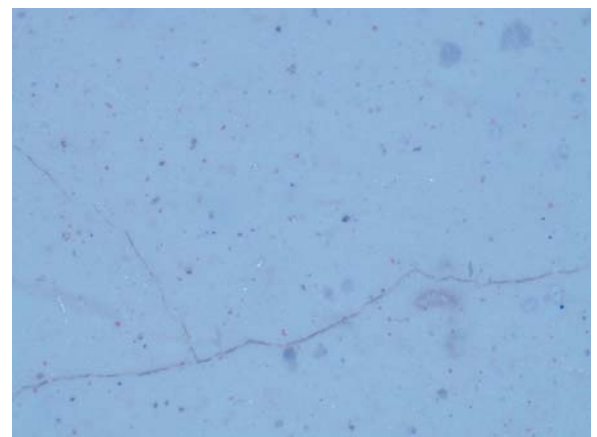
- Very high workability
- No brush marks
- Setting time: 90-100 min.

**Ease of Application**

- Mix is very thin and inconsistent
- Can be easily applied using brush or roller
- Surface Texture: Medium roughness
- Score: 5.0

**Fig. 3.46.2** shows no cracks depressions all over the sample surface**Cracks development**

- Few small size cracks developed on the sample surface. Lots of large surface depressions can be observed all over the sample due to the very thin mix.
- Score: 2.0

**Fig. 3.46.3** digital microscopic picture showing medium size transverse cracks (scale 25 μm)

## **Matrix No. 47**

**Table 47**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	100
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
H <sub>2</sub> O	4
Admixture 2	3.3

### **Workability**

- Low workability
- Very few brush marks
- Setting time: 30-40 min.

### **Ease of Application**

- Mix is very consistent with no lumps
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 2.5

### **Cracks development**

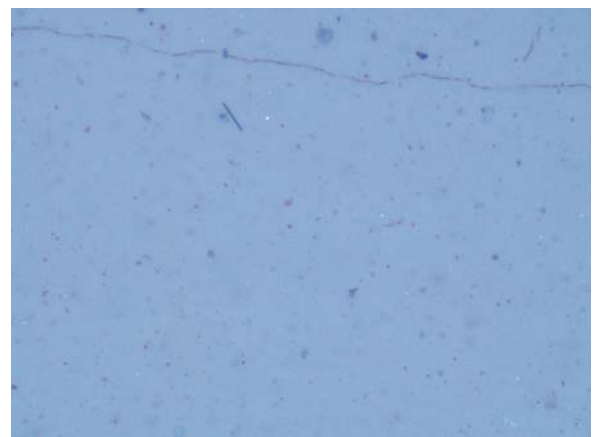
- Very few cracks were developed on the sample surface. No surface depressions were observed.
- Score: 0.5



**Fig. 3.47.1 shows layout of sample mix no. 47**



**Fig. 3.47.2 shows no cracks and smooth surface texture**



**Fig. 3.47.3 digital microscopic picture showing minimal transverse cracks (scale 25 μm)**

## **Matrix No. 48**

**Table 48**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	120
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
H <sub>2</sub> O	4
Admixture 2	3.3



**Fig. 3.48.1** shows layout of sample mix no. 48

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 40-45 min.

### **Ease of Application**

- Mix is very consistent with no lumps
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 3.5



**Fig. 3.48.2** shows no cracks and very smooth surface texture

### **Cracks development**

- Some fine cracks were developed on the sample surface. No surface depressions were noticed.
- Score: 1.5



**Fig. 3.48.3** digital microscopic picture showing small to medium size transverse cracks (scale 25  $\mu$ m)

## **Matrix No. 49**

**Table 49**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	120
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
Fibers 2	1.5
H <sub>2</sub> O	4
Admixture 2	3.3



**Fig. 3.49.1 shows layout of sample mix no. 49**

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 40-45 min.

### **Ease of Application**

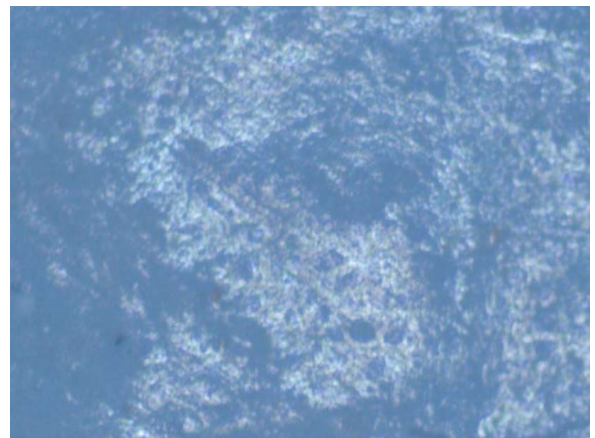
- Mix is very consistent with no lumps
- Can be easily applied using brush or roller
- Surface Texture: very low roughness
- Score: 3.5



**Fig. 3.49.2 shows no cracks and very smooth texture**

### **Cracks development**

- Few cracks are developed on the sample surface. Large depressions were created on different locations which can be clearly seen on Fig. 2.49.3
- Score: 2.5



**Fig. 3.49.3 digital microscopic picture showing no cracks (scale 25 μm)**

## **Matrix No. 50**

**Table 50**

<b>Mix Content</b>	<b>weight (gms)</b>
Part A	100
Part B	120
Part C	10
Micro fiber	15
Ground Filler	30
TiO <sub>2</sub>	5
Fibers	1
Fibers 2	3
H <sub>2</sub> O	4
Admixture 2	3.3

### **Workability**

- Medium to high workability
- No brush marks
- Setting time: 40-45 min.

### **Ease of Application**

- Mix is of consistent with no lumps
- Can be easily applied using brush or roller  
(Two layers were applied on the sample)
- Surface Texture: low to medium roughness
- Score: 3.0

### **Cracks development**

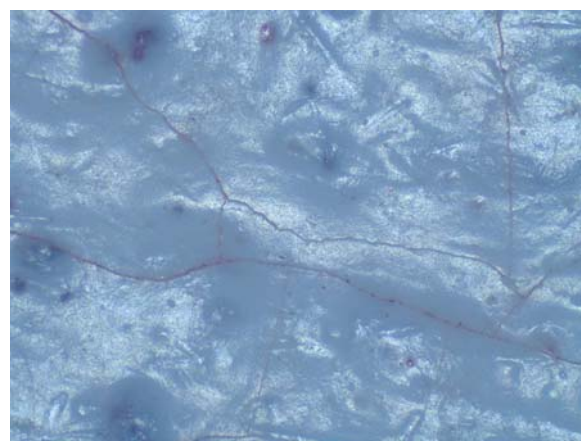
- Lots of large depressions were developed on the fibers locations. Small to medium sized cracks can be seen on many parts of the sample.  
Score:4.5



**Fig. 3.50.1 shows layout of sample mix no. 50**



**Fig. 3.50.2 shows medium size cracks all over the sample surface**



**Fig. 3.50.3 digital microscopic picture showing longitudinal and transverse cracks (scale 25 μm)**

## CHAPTER 4. EXPERIMENTAL INVESTIGATION

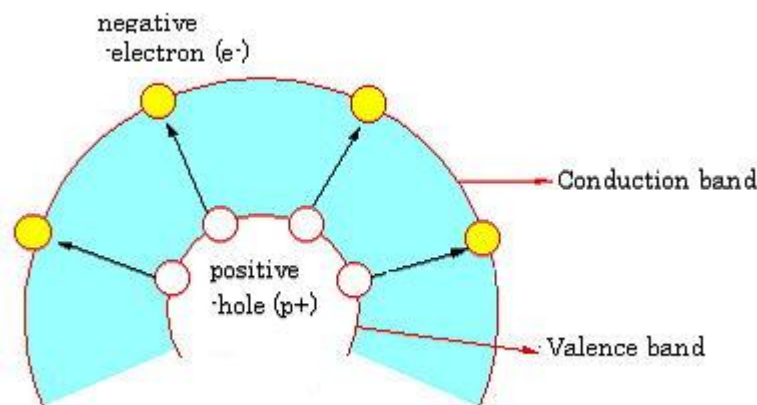
### **4.1 Introduction**

It is well known that the green building movement is all about the efficient use of resources to create and maintain a clean environment. This creates a great interest in the design and development of the so-called “self cleaning concrete coating”, referring to a coating that provides a self-cleaning surface. The achievement of such coatings means, not only a reduction in the problems of pollution and in health risks, but also a reduction or elimination in the consumption of toxic industrial detergents. Achieving these properties on a surface is possible by means of using different coating mixes (Ultimate coating Matrix), and in which nano particles & coarse particles of titanium dioxide ( $\text{TiO}_2$ ) plays a key role. Most of these coatings acquire their self-cleaning capacity through the photocatalytic properties of titanium oxide ( $\text{TiO}_2$ ).

In order to understand the mechanism of the self concrete coating, the photocatalytic behavior of  $\text{TiO}_2$  has to be identified and investigated. This chapter presents the experimental work which was done by applying the self cleaning coatings on cement bricks. Self cleaning properties were recorded through colorimetric measurements of the degradation of the colored dye. The colored dye is composed of organic pollutants which has similar chemical composition like pollutants in the atmosphere.

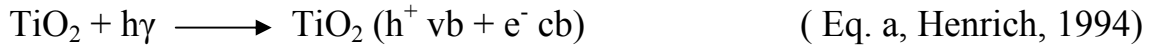
## 4.2 Mechanism of TiO<sub>2</sub> in the Base Mixes

Photocatalytic self cleaning performance has been acquired through the usage of the nano particles & coarse particles of titanium dioxide in the mix. The main philosophy of the TiO<sub>2</sub> photocatalytic action is the exposure of TiO<sub>2</sub> particles to Ultra Violet rays. TiO<sub>2</sub> particles store the light photons of energy equal to or greater than the energy gap which is the region where a particle is forbidden from propagating. The energy stored in TiO<sub>2</sub> particles is sufficient to make an electron excites from the Valence band to the Conduction band. Hence, the movement of the electron will create a positive charge in the valence bond called hole ( $h^+$ ) and the free electron liberated to the conduction band will carry a negative charge ( $e^-$ ). This can be illustrated from the following diagram.



**Fig 4.1 shows the electron excited from the valence band to conduction band (Wikipedia)**





Where:

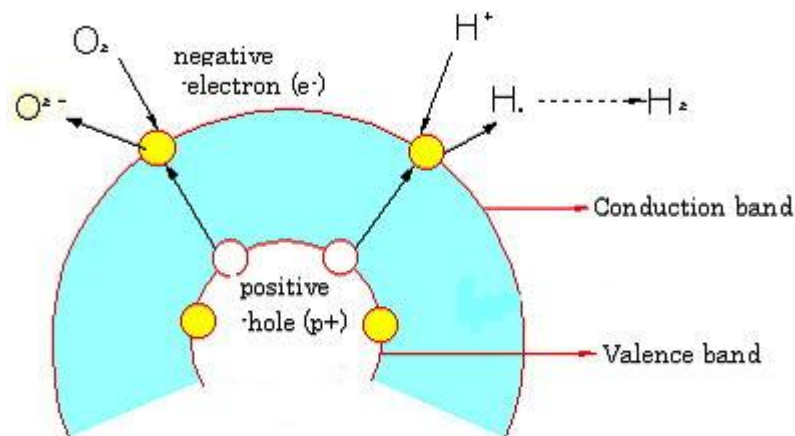
$h$  = Planck's constant

$\nu$  = frequency of Ultraviolet light Rays

$h^+$  = positive charge in the valence band

$e^-$  = free electron liberated to the conduction band.

The extra absorbed energy from light photons will be released as heat. The positive carrier charges or in other words the holes ( $h^+$ ) will react with the surface hydroxyl groups ( $\text{OH}^-$ ) and the surface adsorbed water molecules to form hydroxyl radicals. Radicals are high reactive species which has one unpaired electron. This can be clarified through the following diagram:



**Fig 4.2 shows the formation of hydroxyl Radicals (Wikipedia)**



Where:

$\text{OH}^-$  = The hydroxyl group

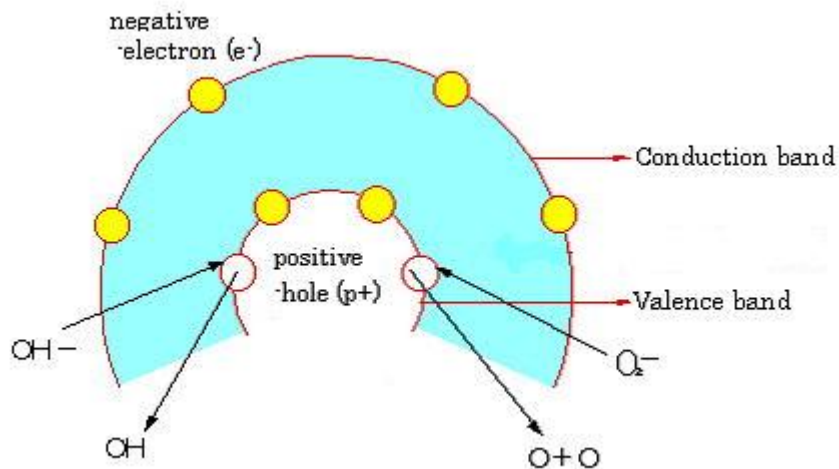
$\text{OH}^\cdot$  = The hydroxyl radical

$h^+$  = positive charge in the valence band

$\text{H}_2\text{O}$  = Surface adsorbed water molecule

$\text{H}^+$  = Hydrogen

Moreover, the negative electron ( $e^-$ ) can react with oxygen ( $\text{O}_2$ ) in the air and super oxide anion ( $\text{O}_2^-$ ) will be formed. The formation of the superoxide will maintain the electric neutrality within the  $\text{TiO}_2$  particle and will prevent it from decomposition. The final result of this process will be hydroxide radicals ( $\text{OH}^\cdot$ ) and hydroperoxyl radical  $\text{HO}_2^\cdot$ .



**Fig 4.3 shows the formation of superoxide anions and hydroxide radicals (Wikipedia)**



Where:

$O_2^{\cdot}$  = Oxygen Radical

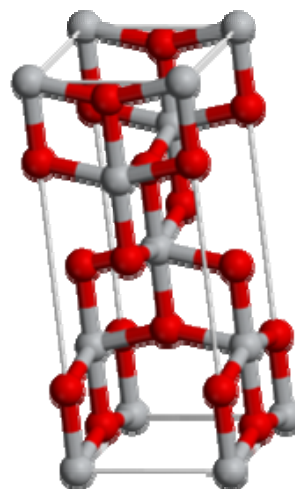
$O_2^-$  = Super oxide anion

$H_2O$  = Surface adsorbed water molecule

$H^+$  = Hydrogen

Hydroxyl radicals are very strong oxidizers and will attack all kinds of organic materials, including organic pollutants resulting in degradation.

The degradation rate of organic pollutants using  $TiO_2$  is dependent on four factors. First, the reaction is affected by the number of light photons on the reaction surface (artificial light versus sunlight). The second factor affecting the degradation rate is the surface area of  $TiO_2$  particles (nano  $TiO_2$  and coarse  $TiO_2$  particles). The third factor is the availability of oxygen to generate hydroxyl radicals which reacts with the organic materials and breaks the bonds between them. The fourth factor to consider is the amount of catalyst particles available and the crystal structure. The photocatalytic activity of  $TiO_2$  is affected by the crystal structure, which in turn controls the energy band gap. Titanium dioxide exists in four crystalline forms which are anatase, rutile, brookite and monoclini



**Fig 4.4 The anatase crystal structure of  $TiO_2$  (Wikipedia)**

c-TiO<sub>2</sub>. In terms of the photocatalytic activity, anatase is more catalytically active than the rutile form. The TiO<sub>2</sub> used on the experiments was in anatase crystalline form.

### **4.3 Sample Preparation and application**

#### **4.3.1 Preparation of Base mixes**

The self cleaning coating is prepared as follows. A mixture of 100 grams of silicate solution and 135 grams of silicate powder is placed in a high-shear mixer containing notched stainless steel blades and mixed for one minute at speed of 1,500 RPM. Few geopolymers particles stick to the wall of the mixer. A putty knife was used to collect the particles sticking to the mixer wall and the mixture is mixed for 1 minute. A mixture of 10 grams of silica fumes, 10 grams of metal oxide, 15 grams of micro fibers, 5 grams of titanium dioxide and 3 grams of fibers is added to the mix and mixed for one minute. Putty knife is used again to collect all the mixture particles sticking to the mixer wall and mixed for one more minute. Ten grams of distilled water is added to the mix and mixed for one minute.

### **4.4 Preparation of Other mixes**

#### **4.4.1 Retarder Mixes**

Same procedure of mixing as in the base mixes is used. The retarder is dissolved in distilled water then added to the mixture & mixed for one minute.

#### **4.4.2 Admixture Mixes**

Same procedure of mixing as in the base mixes is used. Admixture is added to the mixture and mixed for one minute. Then, distilled water is added to the mix and mixed for one more minute.

#### 4.4.3 Glowing Powder Mixes

The glowing powder was added to the mixture during the second step and mixed for one minute. Then, distilled water is added to the mix and mixed for one minute.

Self Cleaning Coating Mix application:

Initially, Self cleaning coating mix is stiff and eventually mixes to a thick liquid that can be applied using brush or roller. The mix was applied on concrete blocks using a smooth brush or a roller. The concrete blocks were cleaned using a piece of cloth before coating application. The concrete blocks dimensions are 24 X 24 X 8 inch . The samples were left at room temperature for at least 21 days before testing.

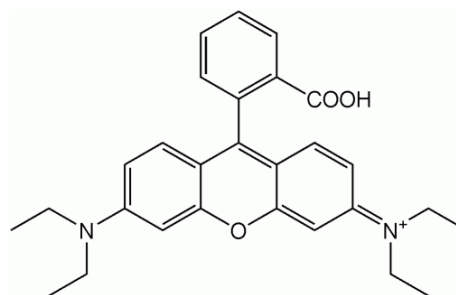
#### 4.5 Curing Method

The coating was cured at room temperature. At room temperature, the sample has to be protected from running water or direct rain for 3 days. After 24 hours, the samples are water resistant. However, running water could damage the surface by leaching out small amounts of mix components.

#### 4.6 Self Cleaning Test Setup

##### 4.6.1 Using Artificial Light

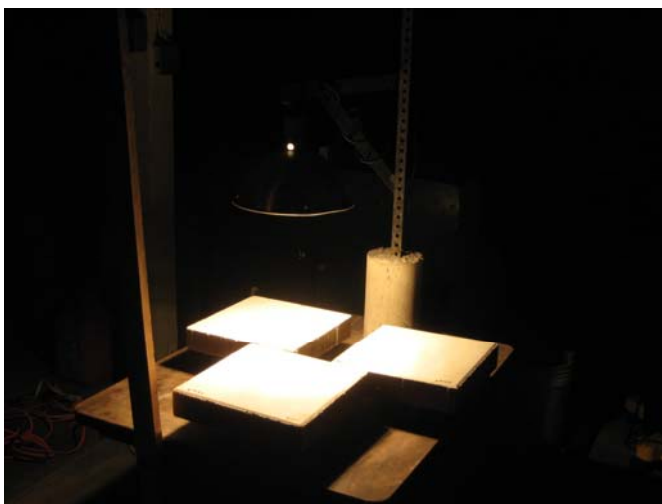
The purpose of this test is to measure the rate of the degradation of the organic pollutants on the self cleaning coating surface. In order to achieve that, an organic dye of rhodamine B is used. Rhodamine B is an organic dye that acts like the organic pollutants



**Fig 4.5 Rhodamine B organic structure (Wikipedia, 2006)**

found in the atmosphere and it is used also as a tracer dye within water to determine the rate and direction of flow and transport. Rhodamine B pigments was added to water to form a red solution. Nine circles of 1.7 inch diameter were drawn using the pencil on the surface of the self cleaning coating. The solution was applied inside the circles using a small brush.

After 24 hours of the dye application, the concrete blocks was exposed to Ultra Violet radiation for 30 hours period and the reading were taken every hour (22 readings were taken during the 30 hours). The UV light source is 300 W lamp placed at a distance of 1.1 Feet from the concrete block. The artificial light source provides a wavelength that is similar to the sunlight. However, the experiments were performed using an artificial light source and sunlight as mentioned later.



**Fig 4.6 shows the concrete blocks subjected to artificial light**

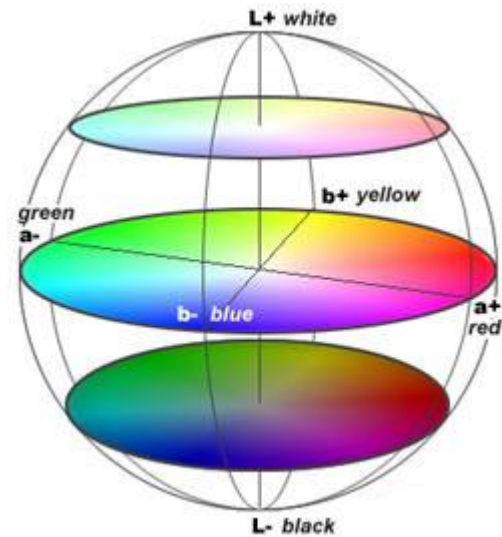
#### **4.6.2 CIELAB color system**

The degradation rate of the organic dye was measured using a Color reader CR-10 (Konica Minolta Sensing colorimeter). The results are shown in form of a table using the CIE (1976)  $L^*a^*b^*$  Color system. The  $L^*a^*b^*$  color system is the most widely used

coloring system for specifying objects colors and calculating color difference. It is also used in textiles, ink, paint and other industries. The CIELAB color system is organizing the colors so that the numeric differences between colors match the visual perception.

The CIELAB system is based on the perception of the colors by the eye and the brain in which the information from eye receptor gets coded in the brain into light to dark, red to green and yellow to blue. Colors cannot be

red and green at the same time, or yellow and blue at the same time. However, colors can be considered as combinations of red and yellow, red and blue, green and yellow, and green and blue. This System facilitated and simplified the communication of color difference information between people worldwide.



CIELAB system Color Coordinates are:

$L^*$  = Lightness coordinate

Where,

$L^+$  = Lighter

$L^-$  = Darker

$a^*$  = the red/green coordinate

**39H**Fig 4.7 shows the CIE Lab coordinates Ref. (UPV.es)



**Fig 4.8** shows the layout of the concrete blocks subjected to artificial light

Where,

$a^+$  = redder

$a^-$  = greener.

$b^*$  = the yellow/blue coordinate

Where,

$b^+$  = yellower

$b^-$  = bluer

#### **4.6.3 Using Sunlight**

The purpose of this test is to measure the rate of the degradation of the organic pollutants on the self cleaning coating surface. An organic dye of rhodamine B was used that acts like the organic pollutants found in the atmosphere. Four circles of 1.7 inch diameter were drawn inside a circular concrete sample of 4 inch diameter and 0.25 inch thickness. The circles were drawn using the pencil on the surface of the self cleaning coating. The solution was applied inside the circles using a small brush.

After 24 hours of the dye application, the circular samples were exposed to Ultra Violet radiation for 48 hours period and two readings were taken before and after the exposure to the sunlight.

The degradation rate of the organic dye was measured using a Color reader CR-10 (Konica Minolta Sensing colorimeter). The results are shown in form of a table using the CIE (1976)  $L^*a^*b^*$  Color system.



#### 4.7 X-Ray Photoelectron Spectroscopy (XPS)

X-Ray Photoelectron Spectroscopy (XPS) or in other words Electron Spectroscopy is a quantitative technique that analyses the chemical composition of the surface of solid materials. X-ray photoelectron spectroscopy works by irradiating the sample placed in an ultrahigh vacuum environment with monoenergetic soft x-rays. The exposure of the sample material to this irradiation causes electrons to be ejected from the atomic shells of the elements at the surface. The energy of these electrons is characteristic of the surface from which they are ejected. Or in other words, identification of the elements in the sample can be made directly from the kinetic energies of these ejected photoelectrons. The number of electrons ejected is counted as a function of energy and the chemical surface composition is obtained.

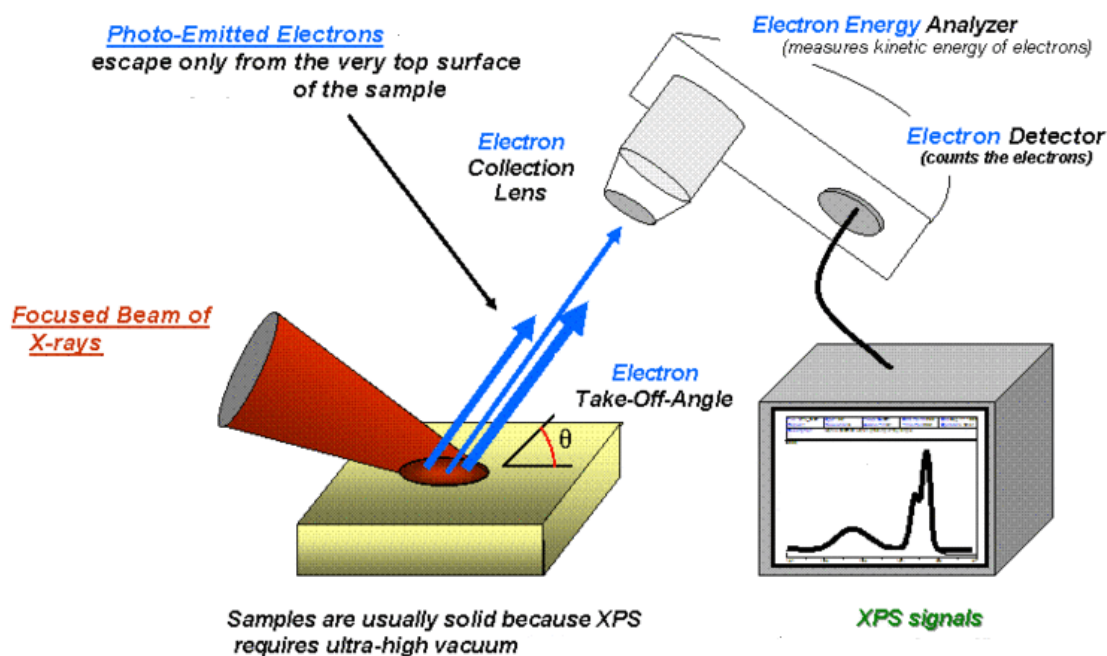
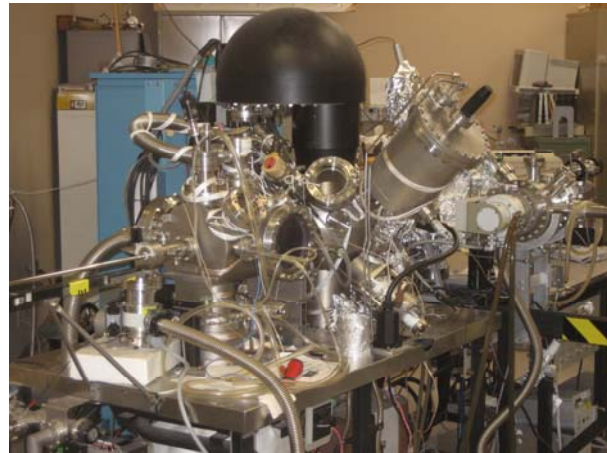


Fig 4.9 show the main components of the XPS instrument (Wikipedia)

**X-Ray Photoelectron Spectroscopy** is composed of the following components:

- Monoenergetic X-Ray source
- Ultra-high vacuum (UHV) steel chamber.
- An electron energy analyzer with an electron collecting lens.
- Magnetic field shielding
- An electron detector system
- A moderate vacuum sample introduction chamber
- Sample mounts
- A sample stage
- A set of stage controllers



**Fig 4.10 shows the XPS in Rutgers Physics Lab**



**Fig 4.11 shows the XPS in Rutgers Physics Lab**

X-Ray Photoelectron Spectroscopy has many capabilities such as the detection of the chemical state of one or more of the elements in the sample, the binding energy of one or more electronic states, identification of the thickness of one or more thin layers (1–8 nm) of different materials within the top 10 nm of the surface. However, the XPS was used in the experimental investigation to determine the depth of TiO<sub>2</sub> in the surface of the self concrete coating. The depth of TiO<sub>2</sub> in the coating plays an important role in the photocatalytic action, hence; it affects the degradation rate of the organic dye and this will be mentioned later on the results chapter.

#### **4.8 XPS Sample preparation**

Five samples of dimension 8mm X 20-30mm X 3mm (approx.) were cut from the self cleaning coating mixes using the electric saw. The samples were then cleaned using piece of cloth to remove all the cement particles during the cutting process.

#### **4.9 XPS testing and experimentation**

The sample is placed in the sample stage for one to ten minutes for a survey scan that measures the amount of all elements. Then, one to ten minutes for high energy resolution scans that reveal chemical state differences. Then, one to four hours for a depth profile that

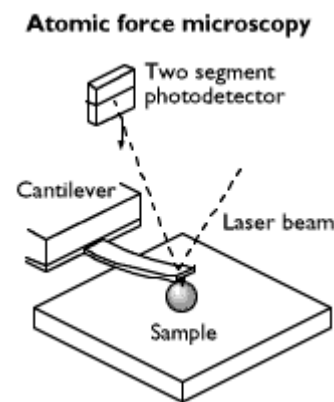


**Fig 4.12 shows the XPS sample dimensions**

measures the Titanium dioxide  $\text{TiO}_2$  as a function of fixed depth. XPS testing accuracy depends on several factors such as the sample homogeneity, accuracy of sensitivity factors, correction for electron transmission function, correction for energy dependency of electron mean free path, and degree of sample degradation due to analysis. However, under the normal testing conditions, the accuracy ranges from 80 to 90 percent which is close enough to determine the element ( $\text{TiO}_2$ ) on the coating surface.

#### **4.10 Atomic force microscope Testing**

Atomic force microscope is a very high-resolution type of scanning microscope, with resolution of fractions of a nanometer, more than 1000 times better than the optical diffraction limit. The AFM consists of a microscale cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature of nanometers. When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law. This tip is exposed to a laser and as it moves on the surface of the sample, the deflection on the tip due to the surface topography is reflected on the laser beam. The laser beam part is connected to a computer that shows the scanned pictures of the sample on the monitor and the roughness average is calculated.

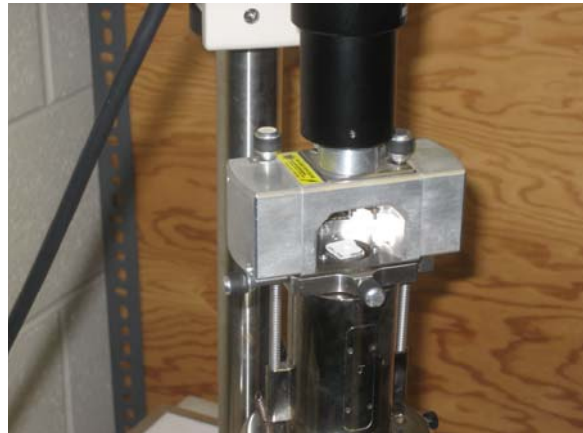


**Fig 4.13 the Mechanism of AFM (Wikipedia)**

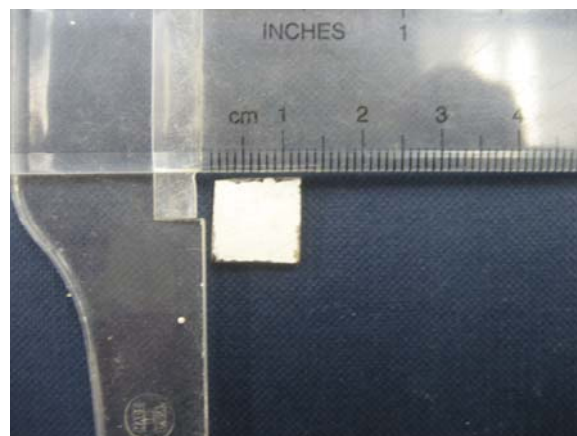
#### **4.11 AFM Sample preparation**

Three samples of dimension 13mm X 15mm X 2mm (approx.) were cut from the self cleaning coating mixes using the electric saw. The samples were then cleaned using piece of cloth to remove all the cement particles during the cutting process.

The AFM testing was done to calculate the surface roughness average. The results of the AFM will be compared to the XPS results to compare the depth profile of Titanium dioxide particles relative to the surface.



**Fig. 4.14 show the sample inside the AFM instrument**



**Fig 4.15 show the AFM sample dimensions**

## **CHAPTER 5. TEST RESULTS AND DISCUSSION**

### **5.1 Introduction**

In this chapter, the test results and discussion of the coating matrix will be presented. The test variables investigated were the classification of the coating mixes according to their workability, ease of application & cracks development (as mentioned in chapter two). In chapter two, each mix was identified solely to show its properties; however, the most efficient mix that can be used in practical applications will be introduced in this chapter. Second, the self cleaning test which was represented by the degradation of the organic pollutant (Rhodamine B organic dye). The decomposition of the organic pollutants can be considered as a breakthrough in the coating mix design. The mix will not only be used as a protective coating but it can be used in many applications such as highways and roads, exterior paintings, kitchens, bathrooms...etc. Third, the depth profile of titanium dioxide was investigated using the X-Ray Photoelectron Spectroscopy machine testing and Atomic force microscope.

### **5.2 Matrix Coating Results:**

From the coating matrix introduced in chapter two, it is obvious that there are lots of mixes that did not develop any cracks or minimal cracks less than one micron. The coating mixes were classified according to the scoring system. From the scoring results introduced in chapter two, the following results can be concluded:

1) Mix 12, mix 13, mix 16, and mix 17 are highly recommended for the coating applications as they have high workability, no brush marks, and setting time of 50 to 60 min. In addition, the mixes are very consistent, can be easily applied using brush or

roller and they have a very low roughness. In addition, no cracks can be observed on the samples surface and very few depressions were developed. The mixes consistency is due to the addition of admixtures which really enhanced the workability and the surface texture of the mix. The setting time was adjusted to be 50 to 60 minutes due to the addition of the retarders. The few depressions that developed on the sample surface were a result of surface depressions. The surface was prepared using a piece of cloth to remove the loose particles. However, there were still lots of surface depressions and holes on the sample surface.

2) Mix 20, mix 21, mix 22, and mix 23 develop almost no cracks or minimal cracks. In addition, the mixes have high workability, no brush marks, and setting time that varies from 55 to 120 min. However, the mixes were very inconsistent, have lots of lumps, and high roughness. The inconsistency of the mix is a result of the addition of retarder 2 was not dissolved in water which leads to the formation of the lumps. Retarder 2 mixes samples are characterized by the glossy surface that they have. Therefore, those mixes can be used on graffiti proof applications.

3) Mixes 35 and mix 36 develop almost no cracks or minimal cracks. Their chemical composition is pretty much similar to mixes no. 12, 13, 16 and 17 except for the silicate powder: fillers ratio. From the chemical composition of those mixes, it can be inferred that slight change in silicate powder: fillers ratio will not affect the coating. However, a big change in this ratio will highly affect the consistency of the mix and will develop lots of cracks.

4) Mix 43 develops very few cracks and is highly recommended for practical applications. However, the addition of retarders and admixture develop lots of cracks. Mix 43 is characterized by its very low roughness and smooth surface.

### **5.3 Self cleaning Results:**

Table 5.1, 5.2 and 5.3 shows the color change rate of the samples for Mix 1, mix 2, and mix 3. The color change is due to the decomposition of the organic because of the photocatalytic effect of Titanium dioxide. This can be clearly illustrated through the graphs of the samples Fig 5.1 to Fig 5.9. A review of the degradation curves mentioned in Figures 5.1 to 5.9 shows the overall effect of nano and coarse titanium dioxide particles on the decomposition of organic pollutants. Those degradation curves present the following:

- 1) The rate of degradation of Rohadamine B organic dye was the highest for Mix B in which coarse titanium dioxide particles were used. This could be due to the fact that the amount of the stored energy of those particles is higher than that stored by nano titanium dioxide particles. Thus, the formation of hydroxyl radicals will be more in mix 2 than in Mix 1 or mix 3 which will increase the degradation rate of the organic dye.
- 2) The curves represented in fig. 5.8 and 5.9 are not linear as the other curves. This is due to the colorimeter reading error. The circles were drawn on the sample surface to minimize that error as they have the same diameter as the Colorimeter lens. However, it is very hard to take readings exactly at the same point which results in some disturbance in the previously mentioned curves.



3) During the testing, procedure, it has been noticed that there was almost no degradation of the organic dye if the dye was applied on a freshly made mix. The curing period of the self cleaning mixes is at least three weeks and after that the organic pollutants can be applied. Fig 5.1 shows the difference in color tones of the dye when it was applied on a fresh mix (48 hours ) and when it was applied after the curing period ( 30 days ). This is due to the suspension of titanium dioxide particles in the mix. Thus X-ray photoelectron Spectroscopy and atomic force microscope were used to investigate the depth of  $\text{TiO}_2$  particles in the mix.



**Fig. 5.1 show the difference in color tones of the dye when it was applied on a fresh mix and after the mix was cured**

4) Mixes 12, mix 13, mix 16 and mix 17 are highly recommended for self cleaning applications as they have high workability, no brush marks, and setting time of 50 to 60 min. The mixes are very consistent and no cracks can be observed on the samples surface. In addition, they have self cleaning properties which can be clearly observed from the degradation of the organic dye.

5) Table 5.5, 5.6, and 5.7 shows the degradation of the organic dye on the sunlight in which the sample were left for 48 hours. The end results show that the degradation in the sunlight is similar to the results under the artificial light.

**Table 5.1**

Sample	Hours	L	a	b
1	1	78.5	21.2	-11.7
2		82.5	13.1	-7
3		78.3	24.2	-12.3
1	2	79	20.7	-10.7
2		82.4	13.3	-6.3
3		78.3	24.8	-12
1	3	80.1	18.8	-9.5
2		82.9	12.7	-5.6
3		79.7	21.7	-9.9
1	4	80.3	18.4	-9.1
2		82.9	12.7	-5.4
3		79.3	21.9	-10.4
1	5	80.6	18.2	-8.3
2		83.1	12.4	-4.7
3		79.7	21.3	-9.4
1	6	80.7	17.6	-7.8
2		83.2	12.3	-4.6
3		79.7	21.6	-9.2
1	7	80.4	16.4	-7.2
2		83.4	12	-4.3
3		80.1	20.7	-8.6
1	8	80.5	17.1	-7.1
2		83.7	11.3	-3.9
3		80.3	20.6	-8.3
1	9	80.7	17.9	-6.9
2		84.8	10.8	-2.9
3		80.8	19.8	-7.7
1	10	81.1	17.1	-6.2
2		84.7	10.8	-2.9
3		81.2	18.6	-5.4
1	11	81.8	16.7	-5.5
2		85.1	9.4	-1.9
3		81.9	17.3	-5.6

Sample	Hours	L	a	b
1	12	81.7	16.9	-5.4
2		85.2	9.3	-1.9
3		82.2	17.3	-5.3
1	13	82.1	16	-5
2		85.4	9.1	-1.7
3		83.2	14.6	-4.2
1	14	81.9	16.9	-5.2
2		85.4	9.1	-1.8
3		83.6	13.9	-3.8
1	15	82.5	15.3	-4.6
2		85.7	9	-1.6
3		83.7	13.1	-3.6
1	16	82.8	15.5	-4.3
2		86.2	8.4	-1.2
3		83.5	14.8	-4.1
1	17	83.4	14.4	-3.8
2		86.2	8.2	-1.2
3		83.9	13.9	-3.6
1	18	83.5	14.6	-2.2
2		87.4	5.7	0.5
3		84.4	13.7	-1.9
1	19	85	11	-1
2		87.5	5.1	0.7
3		84.7	13.2	-1.5
1	20	85.1	11.4	-0.5
2		87.9	4.6	1.1
3		85.7	10.9	-0.5
1	21	84.6	12.1	-2.4
2		88	4.5	0.9
3		84.9	11.3	-2.5
1	22	85.1	11.3	-0.5
2		87.7	4.3	1.1
3		85.7	10.9	-0.5

**Table 5.2**

Sample	Hours	L	a	b
1	1	85.9	8.2	-2
2		86.3	4.9	-1.1
3		86.7	8	-1.3
1	2	87.7	7.9	-1.3
2		87.1	4.8	-0.8
3		88.3	7.8	-2.6
1	3	86.4	7.8	-1
2		87.1	4.4	0.2
3		87	7.7	-0.5
1	4	86.8	7	-0.7
2		87.2	4	0.5
3		87.2	7.3	-0.2
1	5	87	6.8	-0.1
2		87.1	4.2	0.2
3		87.5	7	0.4
1	6	86.9	6.8	0
2		87.3	3.8	0.8
3		87.4	6.9	0.6
1	7	87	6.6	0.1
2		87.3	3.8	0.7
3		87.4	6.6	0.8
1	8	87.1	6.6	0.4
2		87.2	3.8	0.3
3		87.6	6.6	1
1	9	87.1	6.3	0.4
2		87.2	3.9	0.3
3		87.6	6.1	0.8
1	10	87.9	5.4	0.6
2		87.9	3.2	0.4
3		88.9	5.6	2.8
1	11	88.1	4.7	1.4
2		87.8	2.7	1.4
3		88.4	4.9	2

Sample	Hours	L	a	b
1	12	88.1	4.7	1.4
2		87.8	2.8	1.4
3		88.4	4.8	2
1	13	88.1	4.7	1.4
2		87.9	2.8	1.4
3		88.3	4.8	2.1
1	14	88.2	4.4	1.5
2		88	2.7	1.3
3		88.6	4.6	2.3
1	15	88.4	4.3	1.7
2		88	2.5	1.5
3		88.7	4.4	2.4
1	16	88.4	4.2	1.7
2		88	2.5	1.7
3		88.7	4.2	2.5
1	17	89.2	3.1	2.4
2		88.6	1.7	1.9
3		89.6	2.9	3.1
1	18	89.2	2.7	2.4
2		88.6	1.8	1.9
3		89.6	2.7	3.1
1	19	89.4	2.4	2.6
2		88.5	1.3	1.8
3		89.7	2.4	3.2
1	20	89.3	2.5	2.4
2		88.4	1.6	1.8
3		89.7	2.3	3.2
1	21	89.6	1.9	2.7
2		88.7	1.2	2.3
3		90	1.8	3.3
1	22	90.1	1.8	2.7
2		88.9	1.1	2.4
3		90	1.8	3.2

**Table 5.3**

Sample	Hours	L	a	b
1	1	87.8	11.4	-4
2		87.9	11.7	-4.1
3		86.7	9.4	-1.6
1	2	87.7	10.2	-3.7
2		87.1	10.3	-3.6
3		88.3	8.6	-2.4
1	3	87.5	10.6	-3.8
2		87.6	9.3	-2.8
3		88.2	9	-2.5
1	4	88.1	9.5	-3
2		87.7	9.4	-2.9
3		88.3	8.8	-2.3
1	5	88.7	8.6	-2.2
2		87.2	10.5	-3.1
3		88.3	9	-2
1	6	88.4	8.9	-2.4
2		87.1	10.7	-3.2
3		88	9.5	-2.3
1	7	88.3	9.2	-2.4
2		86.6	11.5	-3.6
3		87.9	9.9	-2.4
1	8	88.6	8.6	-2.2
2		86.2	12.6	-4
3		87.9	9.8	-2.2
1	9	88.6	8.6	-2.2
2		86.3	12.3	-3.6
3		88.1	9.2	-2.3
1	10	88.7	8.9	-1.9
2		86.4	11.9	-3.6
3		88.1	9.2	-2.4
1	11	89.1	7.8	-1.1
2		86.9	11.6	-1.9
3		88.7	8.8	-0.5

Sample	Hours	L	a	b
1	12	89.2	7.6	-0.9
2		87.1	11.4	-1.8
3		88.8	8.7	-0.4
1	13	89.4	7.2	-0.7
2		87	12	-1.9
3		88.8	9	-0.5
1	14	89.5	6.9	-0.7
2		87.2	11.5	-1.7
3		89	8.7	-0.4
1	15	89.6	6.9	-0.6
2		87.5	10.9	-1.3
3		89.2	8.2	-0.1
1	16	89.9	6.7	-0.5
2		87.7	11	-1.2
3		89.5	7.8	0.2
1	17	89.8	7	-0.6
2		87.9	10.8	-1
3		89.7	7.5	0.3
1	18	90.4	5.8	0.5
2		89.2	7.9	1
3		90.7	5.2	1.7
1	19	90.8	4.8	0.8
2		90.1	6.3	1.4
3		91	4.6	1.8
1	20	91.1	4.3	1.1
2		90.3	5.8	1.7
3		91.4	3.9	2.1
1	21	91.2	4.2	0.9
2		90.1	6.1	1.2
3		91.2	4.2	1.7
1	22	91.4	3.9	1.4
2		90.2	5.9	1.5
3		91.3	3.6	1.9



**Table 5.4**

Sample	Hours	L	a	b
7	1	82.5	13.1	-7.1
11		85.2	9.3	-1.9
8		86.2	8.2	-1.2

Sample	Hours	L	a	b
7	48	87.1	5.4	0.4
11		87.7	4.4	1.1
8		87.4	4.1	1.3

**Table 5.5**

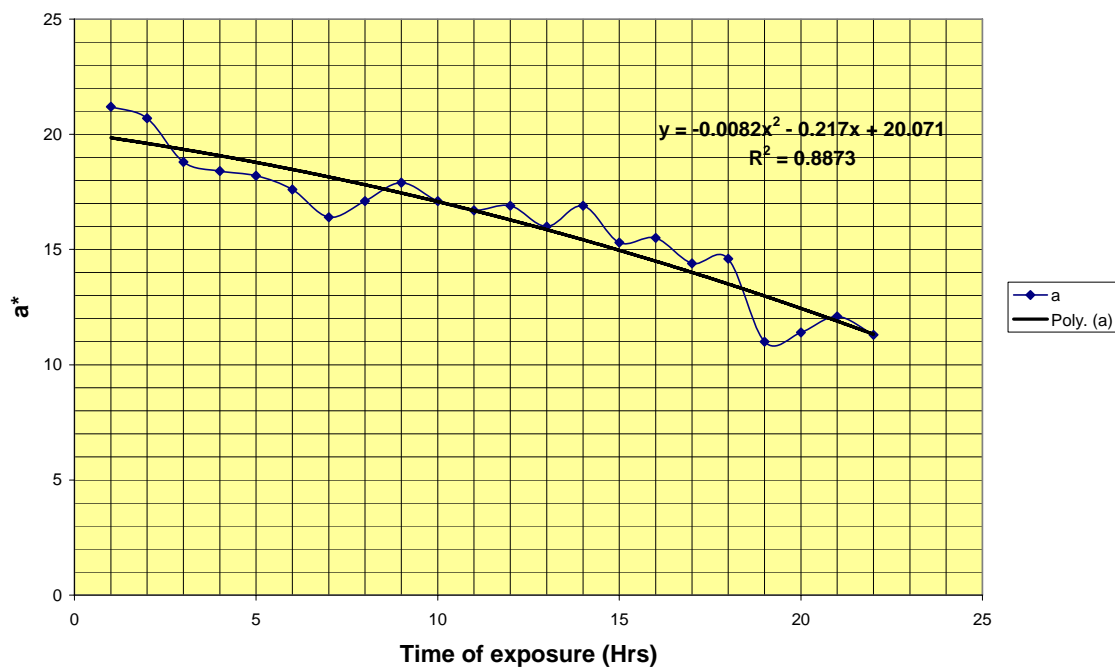
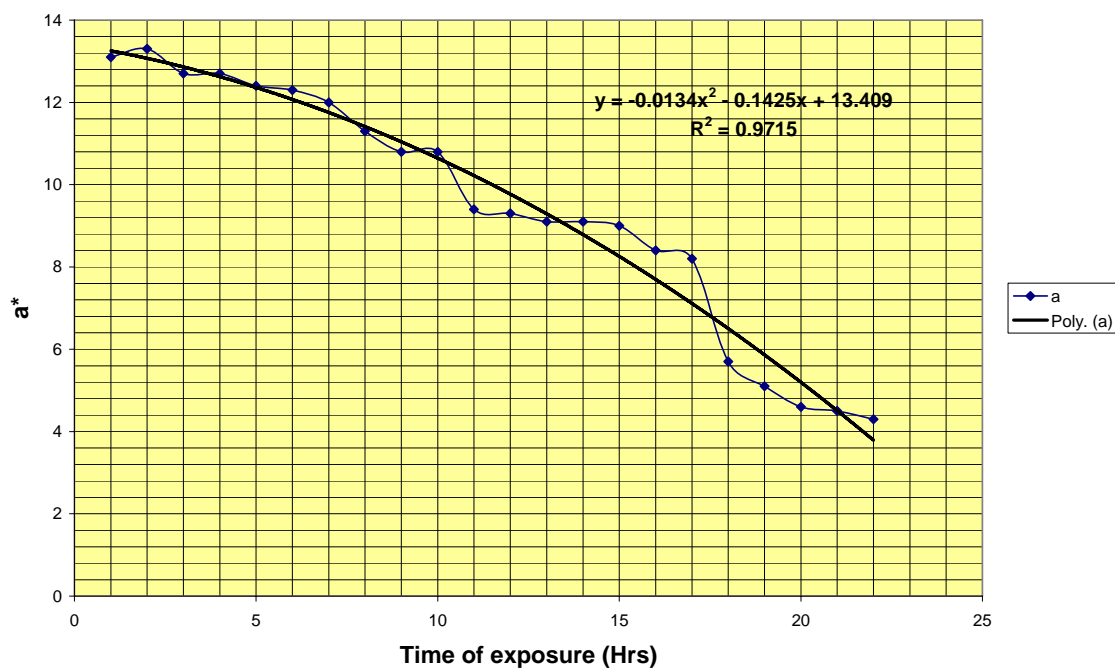
Sample	Hours	L	a	b
7	1	86.8	7	-0.7
11		87.2	6.2	0.5
8		87.2	7.3	-0.2

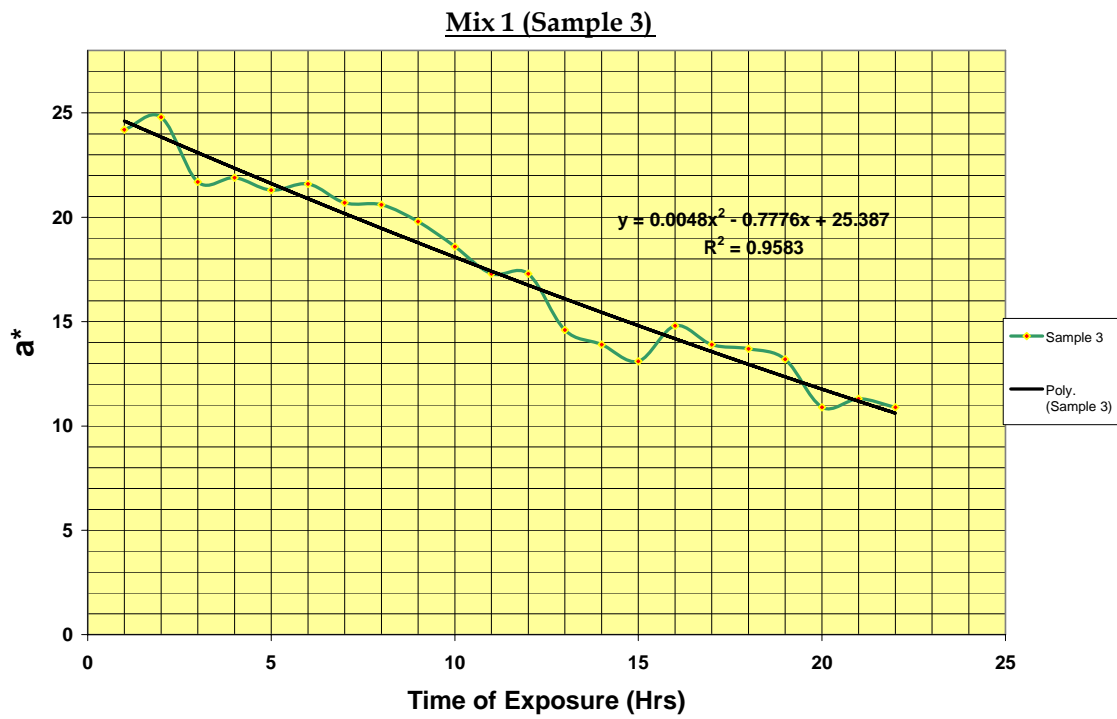
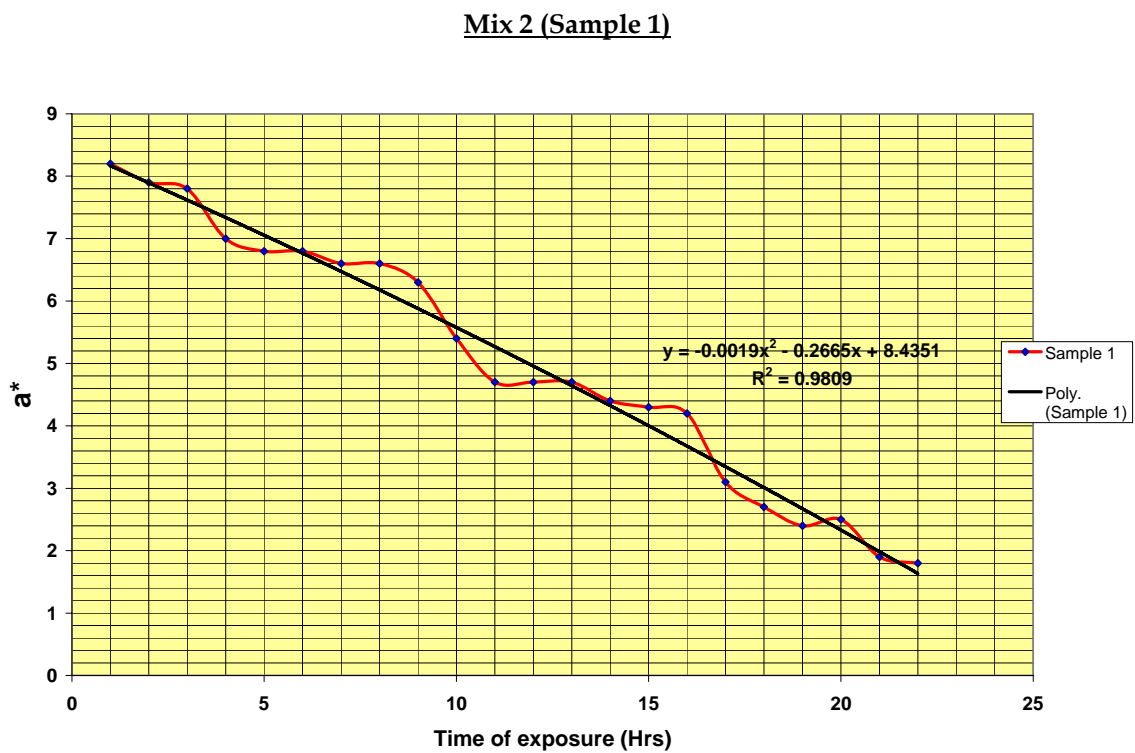
Sample	Hours	L	a	b
7	48	89.2	4.2	2.3
11		87.6	4.3	1.1
8		88.3	4.1	2.2

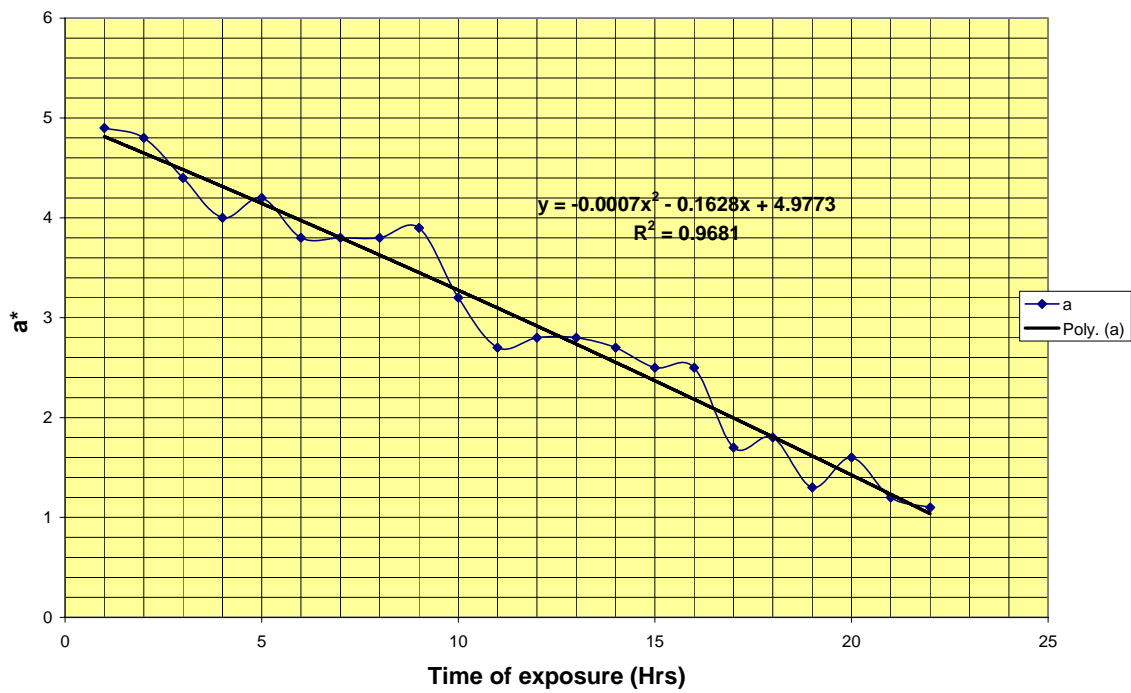
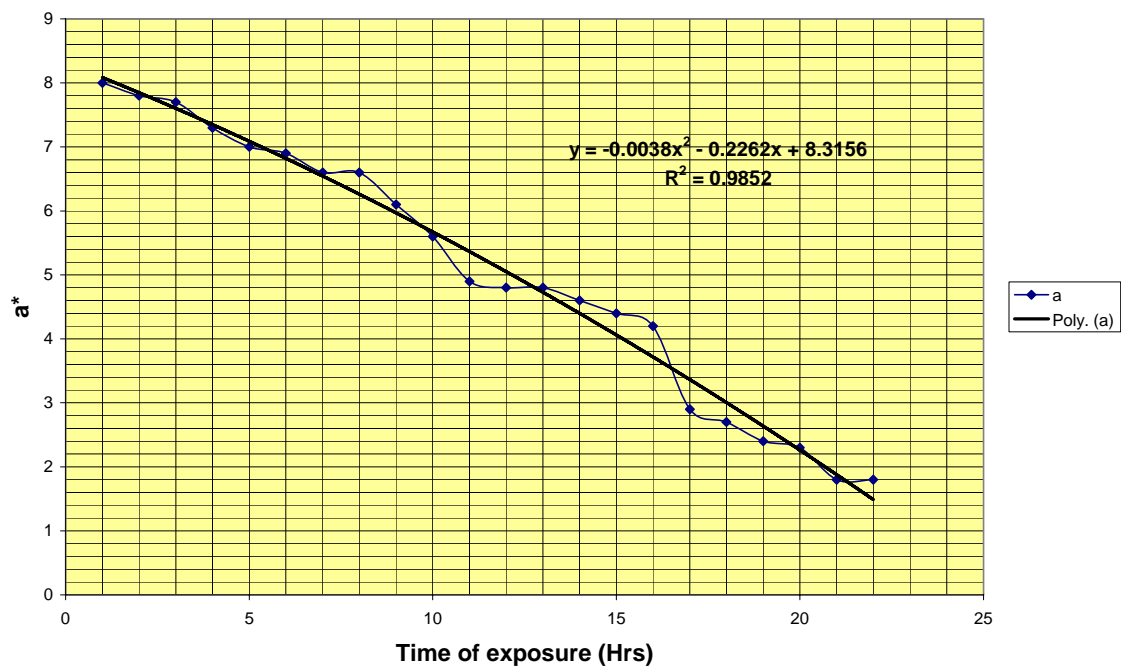
**Table 5.6**

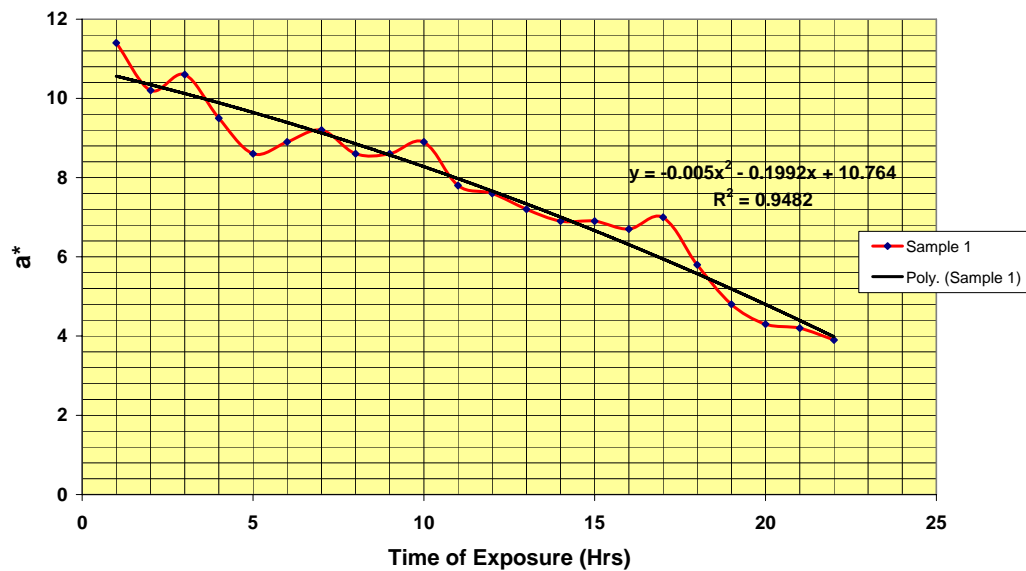
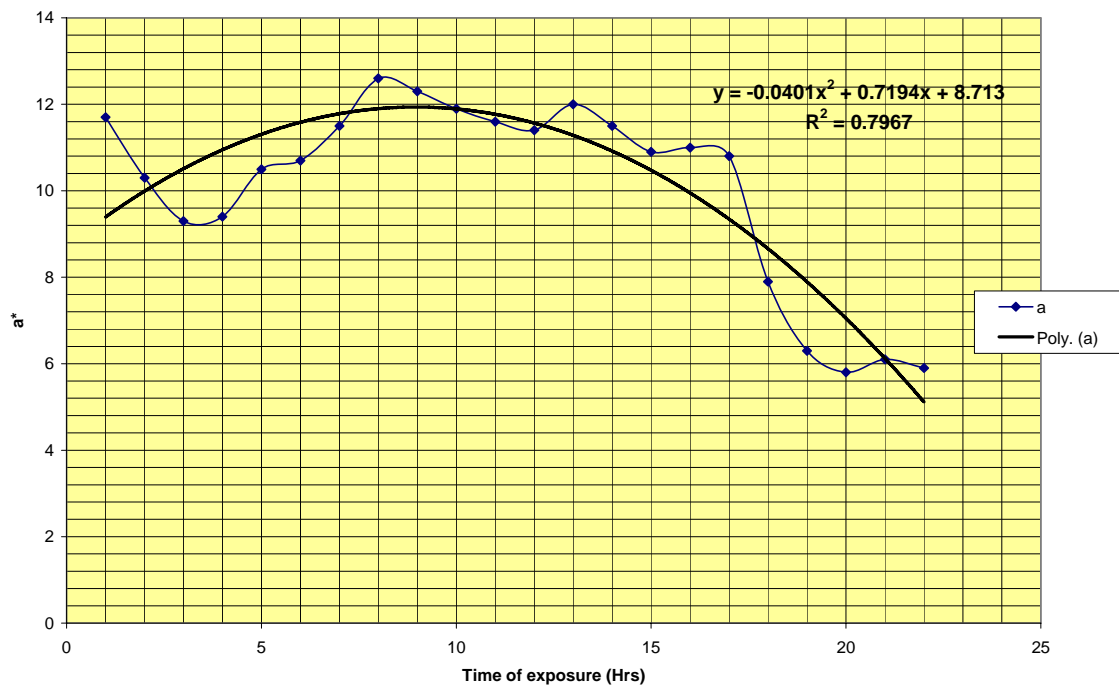
Sample	Hours	L	a	b
7	1	87.1	6.6	0.4
11		87.2	3.8	0.3
8		87.6	6.6	1

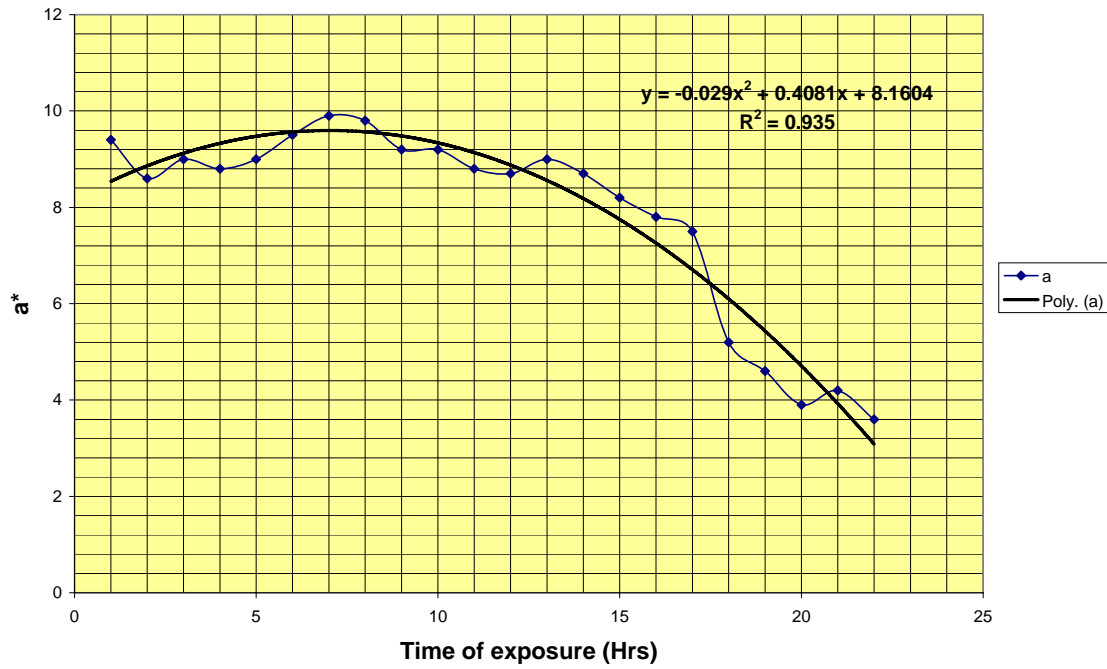
Sample	Hours	L	a	b
7	48	91.4	2.8	1.1
11		91.3	2.9	1.1
8		90.8	2.9	1.6

Mix 1 (Sample 1)Fig 5.1.1  $a^*$  Vs Time exposure for Mix 1 sample no.Mix 1 (Sample 2)Fig 5.1.2  $a^*$  Vs Time exposure for Mix 1 sample no.

Fig 5.1.3  $a^*$  Vs Time exposure for Mix 1 sample no.Fig 5.2.1  $a^*$  Vs Time exposure for Mix 2 sample no.

Mix 2 (Sample 2)Fig 5.2.2  $a^*$  Vs Time exposure for Mix 2 sample no.Mix 2 (Sample 3)Fig 5.2.3  $a^*$  Vs Time exposure for Mix 2 sample no.

Mix 3 Sample 1Fig 5.3.1  $a^*$  Vs Time exposure for Mix 3 sample no.Mix 3 Sample 2Fig 5.3.2  $a^*$  Vs Time exposure for Mix 3 sample no.

Mix 3 sample 3

**Fig 5.3.3  $a^*$  Vs Time exposure for Mix 3 sample no.**

## 5.4 X-Ray Photoelectron Spectroscopy (XPS) and Atomic force microscope

### (AFM) results:

The binding energy of titanium dioxide particles was calculated using the following formulas:

$$E_b = h\nu - E_k - \phi \quad (\text{Einstein Relationship})$$

Where,

$h\nu$  = the X-ray photon energy

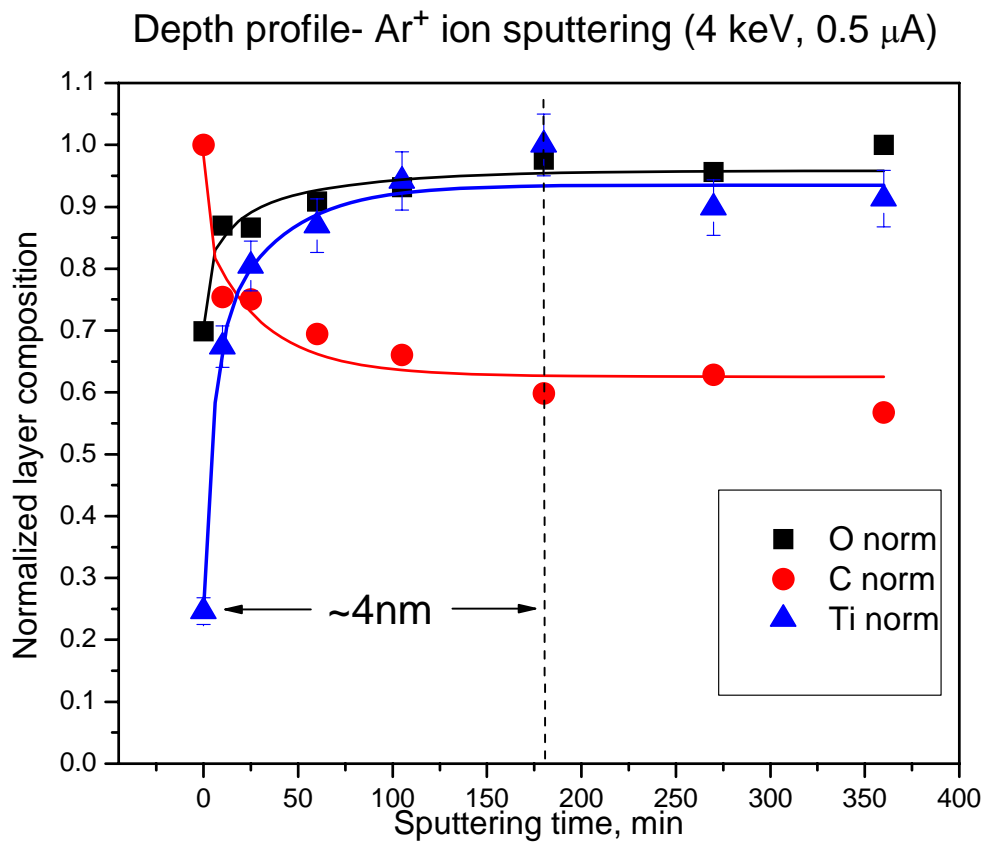
$E_k$  = the kinetic energy of photoelectron (measured by the energy analyzer)

$\phi$  = the work function induced by the analyzer (  $\phi$  can be compensated artificially so it is eliminated).

$$E_b = h\nu - E_k$$

Then, the depth profile was calculated relative to the carbon and oxygen which always exist on the top surface of the sample. Fig 5.2 and fig 5.3 shows the depth profile of titanium dioxide molecules compared to the carbon and oxygen molecules. The average depth profile is equal to 4 nanometers. This average was calculated from the randomly dispersed titanium molecules on the sample compared to the oxygen and carbon molecules. The XPS results was compared to the average roughness obtained from the AFM testing. The average roughness obtained from AFM equals 75.6 nanometer. Fig 5.4 and 5.5 shows the pictures of the sample being scanned using the AFM instrument. Therefore, the titanium dioxide particles exist at the top surface of the sample after the curing period.

These results justify the point that the photocatalytic action of titanium dioxide begins after the curing period of the mix due to the change of depth profile of titanium dioxide particles in the mix.



The estimation is based on attenuation of Ti 2p electron intensity with escaping depth  $\lambda \sim 3\text{nm}$ :  
 $d = 3\text{nm} \times \ln 0.94/0.25 \approx 4\text{nm}$

Fig 5.2 shows the sputtering time Vs Normalized Layer composition



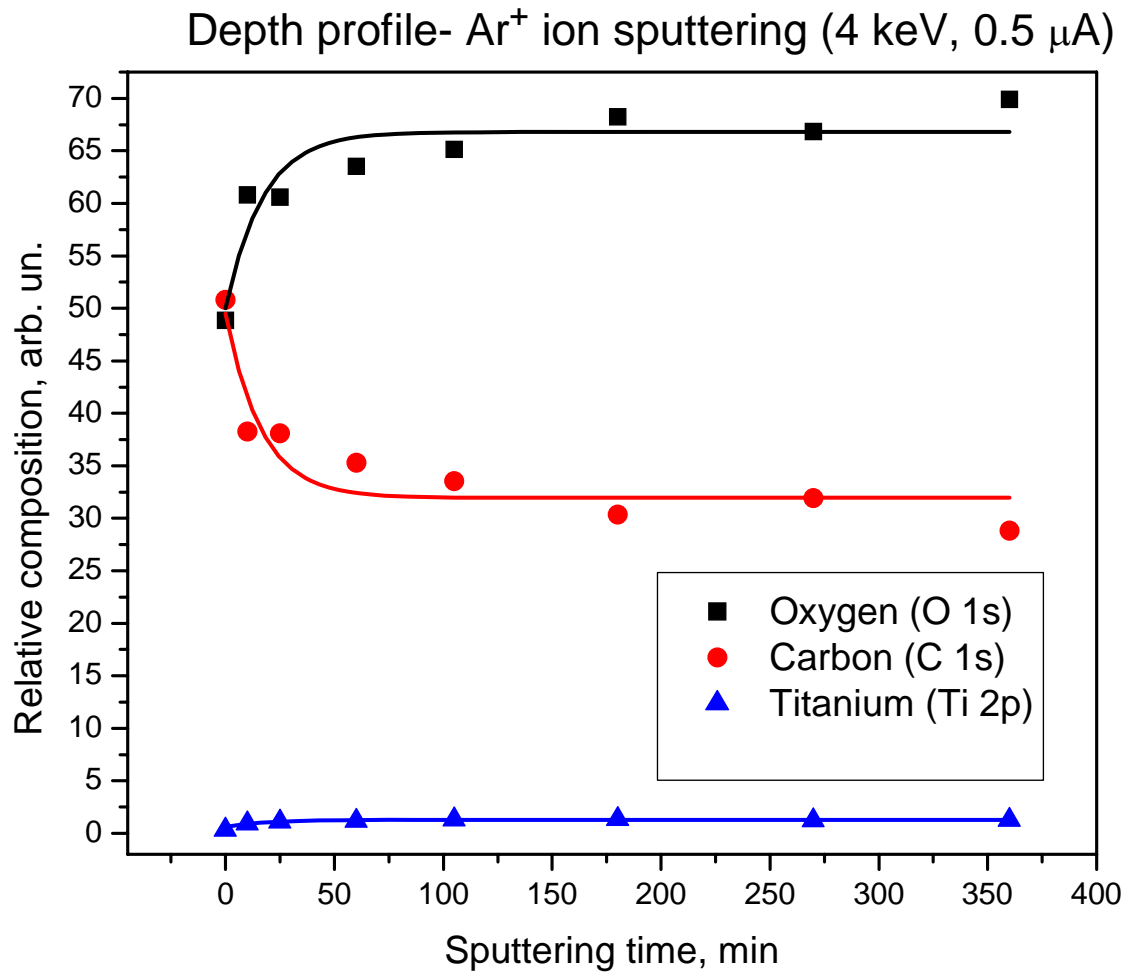
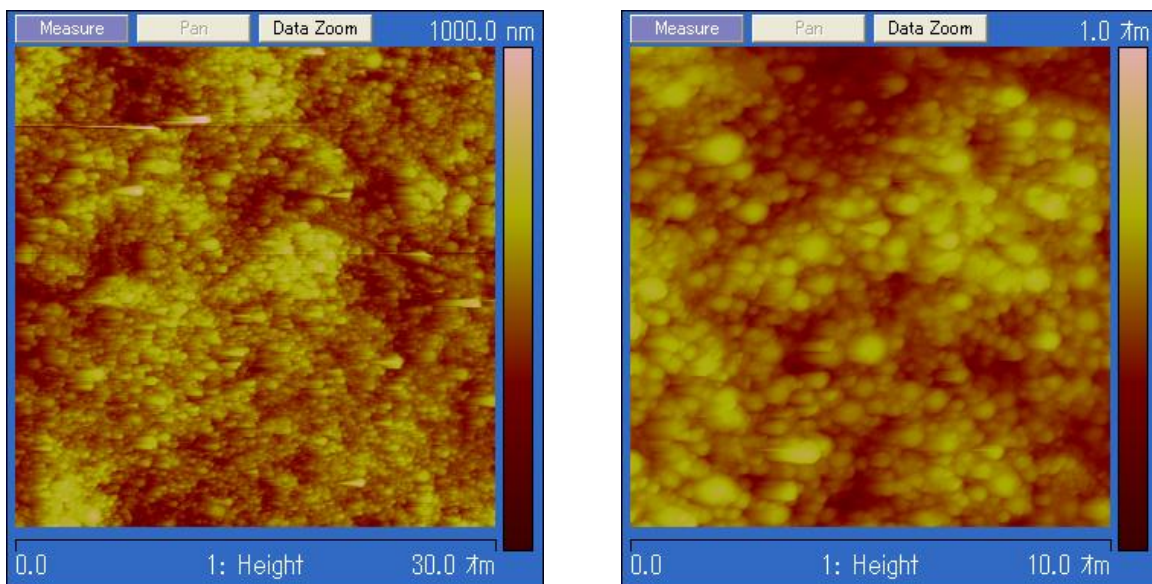
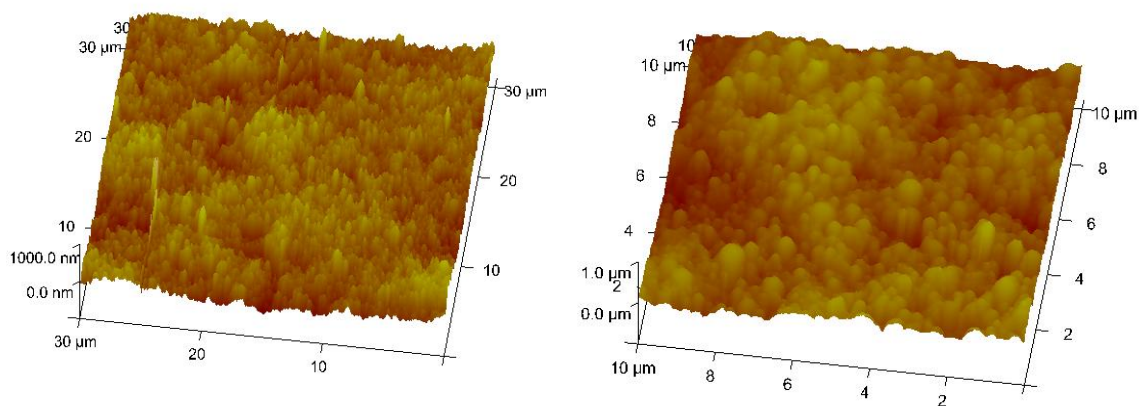


Fig 5.3 shows the sputtering time Vs Relative composition



**Fig 5.4 shows the picture of the sample scanned using the AFM instrument**



**Fig 5.5 shows the 3D model of the sample scanned using the AFM instrument**

## CHAPTER 6. CONCLUSIONS

### 6.1 Conclusions

The results presented in the thesis focus on characteristics and properties of the coating matrix so that it can be used in many applications. Based on the results from the experimental design and experimental investigation, the following points will highlight the significant conclusions:

- The addition of water to the mix is case sensitive because the excess amount of water affects the consistency of the mix and ability of samples to develop cracks. However, the addition of small amount of water will significantly affect the workability of the mix.
- Small change in the Silicate powder: fillers ratio will not affect the coating mix. On the other hand, big change on the previously mentioned ratio will affect the mix significantly.
- The addition of the admixtures plays an important role in the workability and consistency of the mix. The admixtures shall be added within 1% to 2% of the mix weight because the extra addition of the admixture will increase the cracks on the coating.
- The addition of the retarders can be used to improve the setting time. The addition of half a gram to the mix has increased the setting time from 10 min. (approx.) to 60 min. (approx.).
- Retarder 2 mixes are really characterized by their glossy surface which makes them very good mixes for the graffiti proof applications. Nevertheless, the mix consistency and the formation of lumps makes the coating hard to be applied.

- The addition of the glowing powder to the mix gives the coating mix glowing properties and makes it very useful for night applications. Yet, the cracks development on glowing mixes has to be treated.
- The degradation rate of the organic dye is really a breakthrough in the mix design as it gives the coating mix self cleaning properties. Thus, it can be used in roads, highways, buildings exterior and many other applications.
- Titanium dioxide is not used in the photocatalytic reaction (as mentioned before in chapter 3) which will make the coating mix always have self cleaning properties.
- The use of XPS and AFM instruments clarifies the idea of the curing period for the coating mixes. The curing time is a key role in the self cleaning properties of the mix. Thus, the coated surface has to be covered at least for three weeks to make sure that the titanium dioxide molecules are existing at the top surface of the mix and the photocatalytic actions takes place.

## **6.2 Suggestions for Further research**

The following are recommendations for further research in the development of

Inorganic concrete coating matrices and their self cleaning properties:

- Retarder 2 mixes shall be further investigated as they develop no cracks and they have a glossy surface that can be used in graffiti proof applications. However, the inconsistency of the mix and the formation of lumps is the major disadvantage of those matrices.

- The behavior of the self cleaning coating matrices shall be further studied to determine the rate of degradation of organic pollutants using more organic dyes.
- Further studies on the depth profile for freshly made mixes shall be done to determine the suspension of nano titanium dioxide particles in the mixes and the curing phases.

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