

# SYNTHESIS AND CHARACTERIZATION OF WEATHER SHIELD PAINT BY VARYING THE COMPOSITION OF BINDER AND ADDITIVES

By

M. S. Kamal, Rizwan Khan, Irfan Wazeer, Zahid Maqbool, and Shahbaz Ahmad<sup>1</sup>

## Abstract :

Acrylic acid is used as a binder material in the synthesis of weather shield paint. Styrene is also mixed with acrylic acid and resulting compound is used as binder. The effects of varying the composition of additives, binder, solvent and fire retardant have been studied. Fourier Transform Infrared Spectroscopy (FTIR) is used as an analytical tool to observe the changes in the structure of weather shield paint. The rheological behavior of paint is also studied with the help of Rheometer. Thermal analysis has been done with the help of Differential Scanning Calorimeter (DSC). Fire retardancy of paint has been analyzed by performing tests on the paint. By increasing concentration of fire retardant the paint has more resistant towards fire up to a certain ratio of solvent to binder.

**Keywords:** Acrylic, Weather shield, Adhesion, Viscosity, Coating, Rheometer.

## 1. INTRODUCTION

Paints are used for their decorative and protective function. Most basic ingredients of paints are binders, pigments, solvents and additives [1]. An underlying substrate can be deteriorated by the presence of water. A layer of paint can decrease the amount of water at or even in the substrate. The quality of the paint is then also formed by its water transport properties. One of the most desirable aesthetic properties of paint is a high value of gloss and flexibility to encounter cracking during wear [2]. The binder imparts adhesion, binds the pigments together and strongly influences such properties as gloss potential, exterior durability, flexibility and toughness. Binders include synthetic or natural resins such as acrylics, polyurethanes, polyesters, melamine resins, epoxy and oils [3]. Since their introduction decades ago, acrylic polymers have gained a strong foothold in the coatings and allied industries as a result of their improved flexibility and adhesion compared to polyvinyl acetate emulsions, phenolics and styrene-butadiene latex combined with their moderate cost. In addition their significantly improved outdoor durability including resistance to ultraviolet degradation has mandated their use in several applications. Viscosity of paints plays important role in determining its flow behavior hence drying time [4-5]. Different types of flame retardant materials are used in the paints to make paint film flame resistant [6-7]. After their addition to the paints, suitable tests are performed to check the efficiency of the material [8-10]. Internal structure of paint film plays vital role in determining adhesion strength and water barrier properties. Adhesion strength can be increased by adjusting the solvent to binder ratio. The advantages of water-based films and coatings consist in the convenience and environmental safety of their preparation. Especially promising are latex coatings prepared from highly concentrated systems, low-viscous at common temperatures, without using toxic and inflammable organic solvents.

## 2. EXPERIMENTAL

### 2.1. Materials

Basic materials used in these experiments were Ammonia, Acrylic acid manufactured by Khwaja Bashir Ahmed company, Titanium dioxide, Zinc oxide manufactured by Muen Chemical Co. Ltd Microdol, Mergal, Storax, Sodium Hexametaphosphate, Tylose ( $C_{17}H_{32}O_{11}$ ), P 820 (Sodium aluminum disilicon hexaoxide), MTT [3-(4,5-Dimethylthiazol-2-yl)-2,5-Diphenyltetrazolium Bromide], Methyl ethyl glycol, Vegetable oil, Water, Brightly ( $C_{28}H_{20}Na_2O_6S_2$ ), Antimony Trioxide (Flame retardant).

---

1. Department of Polymer & Process Engineering, University of Engineering and Technology, Lahore

## 2.2. Synthesis of paint

The recipe used in the manufacturing process contains; Ammonia 0.3 ml, Acrylic acid 65 g, Titanium dioxide 35 g, Microdol 10 g, Mergal 0.4 g, Storax 0.5 ml, Sodium Hexametaphosphate 0.1 g, Tylose (C<sub>17</sub>H<sub>32</sub>O<sub>11</sub>) 1.5 g, P 820 (AlNaO<sub>6</sub>Si<sub>2</sub>) 5 g, MTT (C<sub>18</sub>H<sub>16</sub>N<sub>5</sub>SBr) 3 g, Methyl ethyl glycol 3 g, Vegetable oil 0.6 ml, Water 80 ml, C<sub>28</sub>H<sub>20</sub>Na<sub>2</sub>O<sub>6</sub>S<sub>2</sub> 100 g. Composition are shown in table 1a & table 1b.

**Table 1a: Composition of paint**

Sample	1	2	3	4
Material				
Acrylic acid (g)	59	65	69	48
Styrene (g)	0	0	0	32
Ammonia (ml)	0.3	0.3	0.3	0.3
TiO <sub>2</sub> (g)	35	35	35	35
Microdol (g)	10	10	10	10
Mergal (g)	0.4	0.4	0.4	0.4
Storax (ml)	0.5	0.5	0.5	0.5
Sodium Hexametaphosphate(g)	0.1	0.1	0.1	0.1
Tylose (g)	1.5	1.5	1.5	1.5
P820 (AlNaO <sub>6</sub> Si <sub>2</sub> ) (g)	5	5	5	5
MTT (C <sub>18</sub> H <sub>16</sub> N <sub>5</sub> SBr) (ml)	3	3	3	3

**Table 1b: Composition of material**

Sample	5	6	7	8
Material				
Acrylic acid(g)	65	65	65	39
Styrene (g)	0	0	0	26
Ammonia(ml)	0.3	0.3	0.3	0.3
TiO <sub>2</sub> (g)	35	35	35	35
Microdol (g)	10	10	10	10
Mergal (g)	0.4	0.4	0.4	0.4
Storax(ml)	0.5	0.5	0.5	0.5
Sodium Hexametaphosphate (g)	0.1	0.1	0.1	0.1
Tylose(g)	1.5	1.5	1.5	1.5
P 820 (AlNaO <sub>6</sub> Si <sub>2</sub> )(g)	5	5	5	5
MTT (C <sub>18</sub> H <sub>16</sub> N <sub>5</sub> SBr)(ml)	3	3	3	3
Methyl ethyl glycol (ml)	3	3	3	3
Vegetable oil(ml)	0.6	0.6	0.6	0.6
Water(ml)	80	80	80	80
C <sub>28</sub> H <sub>20</sub> Na <sub>2</sub> O <sub>6</sub> S <sub>2</sub> (g)	100	100	100	100
Antimony Trioxide(g)	20	25	28	32

First of all solvent is taken in a beaker.

1. An electric stirrer is used in the synthesis of paint. It is immersed in the water and turned on. Then methyl is added to the water and it is mixed in the water until it is fully dissolved in the solvent. It took 5-10 minutes.
2. Sodium Hexametaphosphate is added to the solution. Sodium Hexametaphosphate is used as sequestering agent. It is such a chemical whose molecular structure envelops and hold a certain type of ion in a stable and soluble complex. It is highly effective for collating the paint.
3. Storax NP9 is added and mixed well in paint. Foam is formed due to addition of this material.
4. To avoid foam formation, antifoam is added to the mixture drop wise. Its addition eliminates foam from the mixture.
5. Now tylose is added to the mixture and it transforms mixture into paste. Tylose prevents the sedimentation of coatings and allows almost spatter-free application of the paint.
6. Ammonia liquid is added as a thickener in drop wise fashion. It is added in a small amount to help retard spoilage.
7. P820 (Sodium aluminum disilicon hexaoxide) is added to paint to make surface properties better. It helps paint to give smooth finish.
8. Titanium dioxide is added to paint and it is mixed with the thick paste for 10 minutes. It is white powder and it is a coloring agent.
9. A brightening agent is added now. It is added to increase the gloss of the paint film.
10. Microdol is mixed with the above paste. It acts as a stabilizer against hot and humid conditions. Paint also shows good covering features.
11. MTT [3-(4, 5-Dimethylthiazol-2-yl)-2,5-Diphenyltetrazolium Bromide] is added in the mixture in drop wise fashion and it adjusts viscosity of the paint.
12. MEG is added in the above well mixed paste. It acts as a moisturizer and emulsifier.

Finally temperature of the thick solution is measured and at room temperature binder is added to the mixture. It is allowed to mix for 10 minutes. Then turn off the stirrer and perform quality tests on this paint

### **2.3. Viscosity measurement**

Rheometer was used to measure the viscosity of the paint. Speed of spindle was set at 50 rpm and time interval between readings was set at 10 sec.

### **2.4. Adhesion tests**

Adhesion was tested by making a crosshatch on a dried paint surface. A piece of tape is applied to the crosshatch, and then pulled off.

### **2.5. Weathering tests**

Weathering was tested by exposing the paint to outdoor conditions. The painted surface is exposed to sun, water, and extreme temperature.

## **2.6. Sunlight test**

All samples are coated on a surface and it is placed in open environment. After 72 hours it was observed that color of sample #1 and sample # 2 did not fade. The same coated surface was also examined after 72 hours again and this is repeated for 2 times more and still no change was observed in the gloss and color.

## **2.7. Fire retardancy test**

Fire retardancy was checked by burning the different samples of paint and determining its weight loss.

## **2.8. Structural analysis**

FTIR study was made on JASCO infrared spectrometer. Potassium Bromide was used as an inert background material to get the spectrums.

## **2.9. Thermal analysis of the paint**

SHIMADZU Differential scanning calorimeter is used to analyze paint film thermally. Aluminum is used as reference material

# **3. Result & Discussion**

## **3.1 Adhesion test**

A very small number of particles were observed on the testing tape of sample 1. When tape of sample # 2 was pulled off from the paint, it was compared with the sample # 1 tape and it was clearly seen that even lesser numbers of particles were present on the tape. It showed much better adhesion strength as compared to sample # 1. Result of sample # 3 was compared with the other two and adhesion of this paint film was better than the above two. Actually by increasing the amount of binder, adhesion strength increases. Adhesion tests performed on sample # 4 showed that this new binder has less adhesive strength than the 100 % acrylic resin binder sample. Because sample # 4 contains a binder that is mixture of styrene and acrylic acid, it means this sample has less amount of acrylic acid than all of the other samples so its adhesion decreases.

## **3.2. Weather resistance tests**

Sample #3 was found best amongst all of the others. All samples were allowed to dry off completely and then water test was performed on the coated surface. It was observed that the sample #3 was water repellent and least amount of moisture was found in the film, because it contains higher amount of binder. Although sample # 4 has overall higher percentage of binder but its weather resistance decreases because binder used in sample # 4 is not pure acrylic acid, it is mixture of styrene and acrylic acid as we step down from 100 % acrylic acid as a binder to any lower value a significant decreases is observed in weather resistance and other properties like adhesion. Actually binder is a resin forming a continuous film on the substrate surface. Binders are responsible for good adhesion of the coating to the substrate. The binder holds the pigment particles distributed throughout the coating, more firmly the particles are held more is the resistance to water, sunlight etc.

## **3.3. Sunlight effect**

All samples are coated on a surface and it is placed in open environment. After 72 hours it was observed that color of sample # 1, sample # 2 and sample # 3 did not fade. The same coated surface was also examined after 72 hours again and this is repeated for 2 times more and still no change was observed in the gloss and color. The color did not change because a suitable amount of titanium dioxide is added to the paint during manufacturing and gloss did not change because brightly ( $C_{28}H_{20}Na_2O_6S_2$ ) is added to the paint during manufacturing to maintain gloss. While after 72 hours it was observed that color of sample # 4 faded slightly. Because binder (styrene + acrylic acid) used in this sample has less stronger forces to keep the pigments and additive particles held together in the paint film. That is why sample # 4 paint film has less gloss.

### 3.4. Fire resistance

Result obtained for sample 5,6,7 & 8 are as follow

Table 1: Weight loss for sample 5

Time for burning	Initial weight	Final weight	% weight loss
15	25	23.1	7.6 %
20	25	22.8	8.8 %
25	25	22.7	9.2 %

Table 2: Weight loss for sample 6

Time for burning	Initial weight	Final weight	% weight loss
15	25	23.4	6.4 %
20	25	23.2	7.2 %
25	25	23.0	8 %

Table 3: Weight loss for sample 7

Time for burning	Initial weight	Final weight	% weight loss
15	25	23.6	5.6 %
20	25	23.45	6.2 %
25	25	23.25	7.0 %

Table 4: Weight loss for sample 8

Time for burning	Initial weight	Final weight	% weight loss
15	25	22.5	10 %
20	25	22.0	12 %
25	25	21.35	2.4 %

For sample 5,6 and 7 it was observed that weight loss upon heating was less than 10 %. Any paint that shows weight loss less than 10 % is flame retardant paint. So the experimental results prove that paint that has been manufactured by using the compositions given above for first 3 samples is a flame retardant paint. For sample # 8 weight loss is more than 10% and exceeds the fire retardancy limit. This is because this sample has more amount of liquid materials than all of the other samples. So more evaporation took place and weight loss exceeded 10%.

### 3.5. Viscosity test

Paint samples were tested by rheometer to find viscosity and the results are shown in the figure given below. As the amount of binder is increasing the viscosity is increasing this is because acrylic acid (binder) is a very viscous material and as we increase its amount in paint without increasing amount of solvent, viscosity increases.

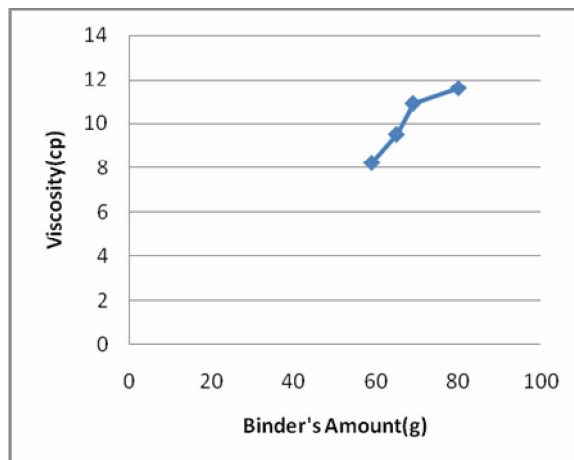


Figure 1: Viscosity as a function of binder concentration

### 3.6. Structural study

Test # 1: A paint sample was prepared having the binder composition 21 %. The structure analysis was done with the help of FTIR.

Area between  $1600-4000\text{ cm}^{-1}$  is referred to functional group region. We can easily see in figure 1 that there are too many peaks present in this region. These peaks are referring to stretching motions of functional groups. Acrylic acid functional group ( $\text{COO-R}$ ) lies in this region. The region between  $500-1600\text{ cm}^{-1}$  is called fingerprint region. In this region peaks normally overlap and mathematical deconvolution is needed to identify groups. The region between  $500-700\text{ cm}^{-1}$  shows the presence of inorganic filler in the paint sample. From  $2400-2200$  absorbance decreases very rapidly that shows presence of stabilizer functional group in this region while other regions show high absorbance values. It means that paint has a greater ability to protect the surfaces from light effects.

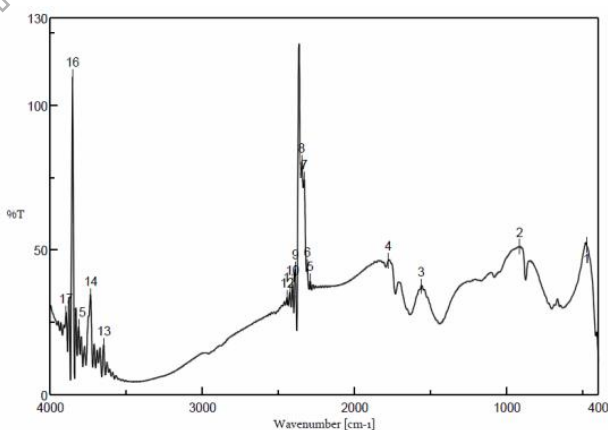
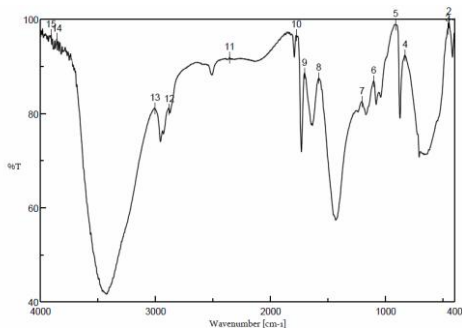


Figure 2: Structural changes due to addition of acrylic acid

Test # 2: A paint sample was prepared having the binder composition 15 %. Different spectra are taken during manufacturing of paint to observe the effect of binder, titanium dioxide and ammonia on the properties of the paint.



**Figure 3:** Structural Changes due to addition of titanium dioxide and ammonia

This spectrum was taken just after addition of binder material. The region 2500-1900  $\text{cm}^{-1}$  shows a broader spectrum and it shows that absorption of light is very consistent in this region. It means extender functional groups are present here. Its consistency shows that extender does not have any effect on absorbance of light so these also don't affect color of the paint.

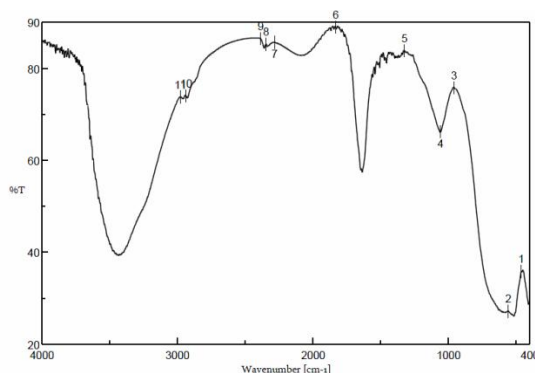
By comparing the above two spectrums it is very clear to us that when concentration of binder and extender was greater in the paint then more broader regions were found in the spectrum. This means that higher the concentration of extender and binder is better the above mentioned properties are.

### 3.6.1 Effect of binder

We performed 2-3 experiments by changing ratio of solvent to binder from 1.25 to 1.12. When Solvent to Binder ratio was 1.25, adhesion tests were performed, it was observed that paint film has acceptable adhesion strength. When this ratio was decreased to 1.23, even better adhesion was observed. Another sample was tested having solvent to binder ratio 1.27 and this time adhesion strength was decreased as compared to above two samples. So it can be concluded from above discussion that upper limiting ratio of solvent to binder lies in the range of 1.23 to 1.25. One sample was prepared having a binder made from mixture of styrene and acrylic acid; it produced less adhesion strength than the paint having 100 % acrylic acid.

### 3.6.2 Effect of titanium dioxide

A series of experiments are performed by changing the concentration of titanium dioxide in the paint from 10 % to 13 %. It is found by increasing the concentration of titanium dioxide, the brightness of paint increases. Titanium dioxide is resistant to heat and solvent used in the manufacturing of paint. We used rutile grade of  $\text{TiO}_2$  because it has high refractive index than anatase grade. Another reason to use rutile grade is that when anatase grade  $\text{TiO}_2$  used in the paint, the resulting product will show chalking. Chalking is due to oxidation of the presence of  $\text{TiO}_2$ . The rutile grade is less active than the anatase grade that is why it does not cause breakdown in the film surface leading to the chalking. The following spectrum is obtained after the addition of titanium dioxide in the paint during manufacturing.



**Figure 4:** Structural changes due to addition of titanium dioxide

A broad band was observed at 3400  $\text{cm}^{-1}$  for as-grown films, which might be due to the stretching of water and hydroxyl groups. The broad band at 550  $\text{cm}^{-1}$  represents stretching of Ti-O bond. The peaks in the region 1200-1500  $\text{cm}^{-1}$  belong to Ti-OH stretching and Ti-O-C Bonding.

#### 4. CONCLUSIONS

- Adhesion strength increases as the concentration of binder increases.
- Water resistance also increases as the concentration of binder increases.
- Fire retardancy is found to be function of concentration of fire retardant.
- It is found by increasing the concentration of titanium dioxide, the brightness of paint increases.

#### 5. ACKNOWLEDGEMENT

The authors are thankful to the Department of Polymer And Process Engineering, University of Engineering and Technology Lahore Pakistan for the Laboratory support.

#### 6. REFERENCES

- [1] Dwight G. Weldon, Failure Analysis of Paints and Coatings, 2009, pp 1
- [2] A.A Tracto, Coatings Technology Handbook, 2005, pp 46-1
- [3] Hans J.Geelhaar, Frankenthal; Erich Penzel, Ludwigshafen' Gregor Ley, Wattenheim, US patent # 4267091, 1981, pp 1-3
- [4] A.C. Aznar, O.R. Pardini, J.I. Amaly, Glossy topcoat exterior paint formulations using water-based polyurethane/acrylic hybrid binders, 2005, pp 1-5
- [5] Paint and coating testing manual, fourteenth edition of the Gardner-Sward, 1995, pp 800-860
- [6] Gunduz G, Kisakurek D, Kayadan S, Flame retardant alkyd paint, 1999, pp 1-6
- [7] Chuen-Shii Chou, Sheau-Hong Lin, Chin-I Wang, Preparation and characterization of the intumescent fire retardant coating with a new flame retardant, 2008, pp 1-4
- [8] W. M.Morgans, Outlines of paint technology third addition, 2000, pp 423-432
- [9] G. G. Sward, Paint testing manual, thirteenth edition, 1972, pp 740-760
- [10] William C. Golton, Analysis of paints and related materials: current techniques for solving coating problems, 1992, pp 126-145
- [11] S. S. Ivanchev, V. N. Pavlyuchenko, N. A. Byrdina, M. Skrifvars, and J. Koskinen, Water-Resistant Films and Coatings Based on Cross-Linking Styrene3Acrylate Latex Copolymers, 2000. PP-1