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Experimental Studies on Sol-Gel Derived Titanium Dioxide Finishing of Fabric For Self Cleaning

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Abstract: *Titanium oxide (TiO_2) in the anatase structure has been used as an excellent photo catalyst for photo decomposition and solar energy conversion. The sol-gel process includes the analysis of inorganic networks through the formation of a colloidal suspension (sol) and gelation of the sol form a network in a continuous liquid phase (gel). The Titanium oxide (TiO_2) sol-gel is prepared by three methods- using acetic acid and solvent ethanol, using nitric acid and solvent water and by acetic acid, solvent ethanol and hydrogen peroxide. All samples deep coated. The fabric samples were dried at 80°C for 10 min and then cured at 100 °C for 10 min in a preheated oven. Finally, steaming treatment was given to the fabric at 97 °C for 1.5 hr to induce the formation of electro-gel TiO_2 i.e anatase. Color Eye 7000Å spectrophotometer is used to know the photo-degradation of stain. Nitric acid hydrolyzed sol-gel TiO_2 treated fabric samples show good stain loss as compared to acetic acid hydrolyzed sol gel TiO_2. Nitric acid hydrolyzed sol-gel TiO_2 treated fabric samples observed under microscope show uniform distribution coating on treated fabric sample. Physical testing of fabric samples show that one has to compromise on draping qualities and service life of the fabric to make fabric self-cleaning.*

Key Words: *Titanium oxide-TiO2, Sol-Gel, Titanium Tetra-isopropoxide, Self-cleaning, Photo catalytic activity.*

1. Introduction

The sol-gel derived Titanium oxide TiO_2 coated fabric has potential applications in biological and chemical protective clothing for health care, food processing and farm workers, as well as military personnel. Reactive oxygen species in coating, such as hydroxyl radicals and hydrogen peroxide, kills bacteria and break down organic compounds such as pesticides and other toxins. Domestic applications of such fabric are wearing purpose, curtains, table cloths etc.

1.1 Titanium and Sol-gel derived TiO_2

Titanium is a chemical element in the periodic table having the symbol Ti, atomic number 22, and atomic weight 47.90. The element burns in air when heated to give the oxide, TiO_2. Titanium occurs in nature as rutile (tetragonal TiO_2), anatase (tetragonal TiO_2), brookite (rhombic TiO_2). Titanium is well known for its excellent corrosion resistance almost as resistant as platinum, being able to withstand attack by acid, moist chlorine gas, and common salt solutions. TiO_2 in the anatase structure has been used as an excellent photo catalyst for photo decomposition and solar energy conversion due to it high photo activity. Generally, anatases have higher photo catalytic activity than rutile.

1.1.1 Sol-Gel

The synthesis of TiO2 nano-particles can done by different ways such as sol-gel, sol, hydrothermal and solvo thermal methods. TiO2 nano-particles apply on textile substrates with the low-temperature methods for the synthesis of highly crystalline TiO2 nano-particles . The development of TiO2 nano-particles application assures retaining desirable mechanical and comfort properties of textile and also enable to obtain highly durable, comfortable self-cleaning textiles, with de contamination rates in time scale of minutes, in the visible spectrum range. The sol-gel process is a wet-chemical technique widely used in the fields of material science and ceramic engineering. In general, the sol-gel process involves the evolution of inorganic networks through the formation of a colloidal suspension (sol) and gelation of the sol form a network in a continuous liquid phase (gel).

1.1.2 The mechanism between TiO_2 nano-particles and cotton fabric

When light is radiated on fabric, if illuminated light energy higher than its band gap, electrons in sol-gel TiO2 will jump from the valence band to the conduction band, and the electron (e) and electric hole (h+) pairs will form on the surface of the photocatalyst, released electrons bind with oxygen to become super oxidized anion. Moreover, surface of fabric becomes positively charged and takes electron from moisture of the air. The moisture that has lost electron forms hydroxyl radical. Then by super oxidized anion and hydroxyl radical's strong power of oxidation destruction, decomposition is generated on surface of fabric. This makes decomposition of stain, dirt etc. and disperse Co_2 in atmosphere.

II. Experimental Work

1.2 Procedure

The experiment is carried out by three different methods as follows:

- By using acetic acid and solvent ethanol
- By using nitric acid and solvent water
- By using acetic acid, solvent ethanol and hydrogen peroxide

The titanium- tetraisopropoxideTi(O-iPr)4 was hydrolyzed by using glacial acetic acid in first & third method. To this solution, the required amount of ethanol (with hydrogen peroxide H2O2 in third method) was added drop by drop under vigorous stirring for 2 hr and the stirring was continued until a clear solution of TiO_2 nano-crystals was formed.

In second method, the titanium- tetraisopropoxideTi(O-iPr)4 was hydrolyzed by using nitric acid and water is added at room temperature drop by drop under vigorous stirring until a clear solution of TiO_2 nano-crystals was formed. These crystals are heated at a heating rate 1°C to a temperature of 500°C in an oven under ambient condition maintaining at this temperature for 30 min. All samples are deep coated and dried at room temperature.

In second method, using water as solvent, after dip coating, fabric was dried at room temperature, rinsed with 1g/ liter soda ash solution and water for neutralization of acid. The fabric samples were dried at 80°C for 10 min and then cured at 100 °C for 10 min in a preheated oven. Finally, steaming treatment was given to the fabric at 97 °C for 1.5 hr to induce the formation of anatase.

2.2 Chemical composition of the methods

In these methods, titanium- tetraisopropoxideTi(O-iPr)4, ethanol and acetic acid were maintained in a molar ratio of 1:100:0.05 respectively as mentioned in Table No. 1 the quantity of chemicals were taken for experiment.

Table No. 1: Chemical Composition of Various Methods

Sr. No.	Chemical composition	Method-I Quantity in ml	Method-II Quantity in ml	Method-III Quantity in ml
1	Titanium Tetra Isopropoxide	02.90	13.00	03.00
2	Ethanol	46.00	NA	46.00
3	Acetic acid	00.03	NA	00.03
4	Nitric acid	NA	003.00	NA
5	Water	Not Applicable	200.00	NA
6	Hydrogen Peroxide	NA	NA	06.00

2. Evaluation

Evaluation of self cleaning activity on fabric can be done as

- Effect of method of preparation and hence the characteristics of nano-particles
- Time of exposure

The self-cleaning action /photo-activity of the TiO_2 coated cotton fabric can be investigated by exposing the samples containing adsorbed stain to visible irradiation. The measured quantity of stain solution is introduced on the cotton fabric and is allowed to spread. Half of stained fabric is exposed to the sunlight for 3-6-9 hr, while the other half is covered with a black paper to prevent its irradiation from sunlight. The exposed part of the stained fabric is compared with that of the covered part for self-cleaning action.

Color Eye 7000Å spectrophotometer is used to measure the photo-degradation of stain. The self-cleaning action is quantified by comparing K/S values (absorption to scattering coefficient) of the exposed and unexposed portions of the same stain by spectrophotometer. The K/S value of unexposed part of the stain was taken as 100 and relative decrease in K/S value of exposed part was calculated using the following relationship:

$$\%\ \text{decrease in K/S of exposed part} = \frac{\left(\frac{K}{S}\right) unexposed - (K/S) exposed}{(K/S) unexposed} \times 100$$

III. Result & Discussion

3.1 Observations

The coated fabric samples were stained by one of the solutions e.g. 10% coffee solution, 20% tea solution or 10% dye solution. The samples were exposed to sunlight for 3, 6 & 9 hrs. The results are as follows:

Method-I

Acetic acid hydrolised sol-gel TiO_2 treated Cotton fabric samples stained by 10% dye solution and exposed to sunlight for 3hrs show 28% stain loss. When exposer continued up to 6 hrs, sample had shown 38% stain loss and further continuation of exposer up to 9 hrs shown 45% stain loss.

Method-II

Nitric acid hydrolised sol-gel TiO_2 treated fabric samples stained by 10% dye solution and exposed to sunlight for 3hrs show 38% stain loss. When exposer continued up to 6 hrs, sample had shown 45 % stain loss and further continuation of exposer up to 9 hrs shown 52 % stain loss. PC blend treated fabric samples were stained by 20% tea solution & 10% coffee solution and exposed to sunlight for 3 hrs had shown 30 % and 35 % stain loss respectively.

Method-III

Cotton fabric sample treated by Sol-gel TiO_2 prepared by using acetic acid, solvent ethanol and hydrogen peroxide were stained by 10% dye solution. They exposed to sunlight for 3hrs had shown 30 % stain loss, continued exposer up to 6 hrs, sample had shown 37% stain loss. When same samples were stained by 20% tea solution & 10% coffee solution and exposed to sunlight for 3 hrs had shown 30 % and 38 % stain loss respectively.

3.1 *Microscopic observations*

We have observed coated fabric samples under microscope with magnification 100 X 100. It is observed that method- II shows uniform distribution coating on treated fabric sample than method-I and method-III.

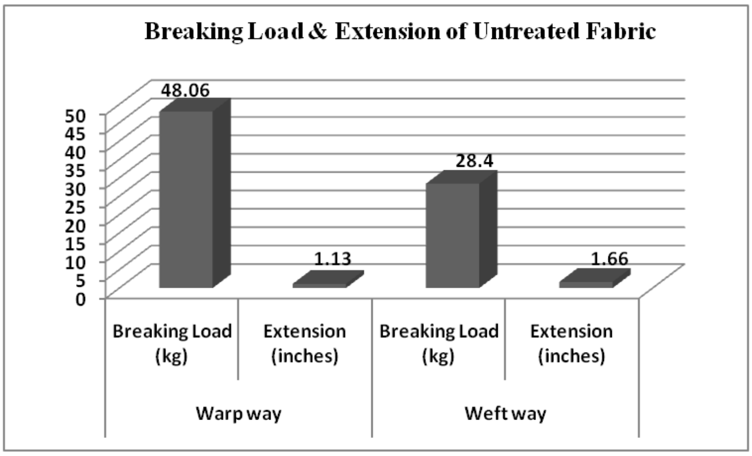
3.2 Physical Testing

Nitric acid hydrolyzed sol-gel TiO_2 treated fabric samples show even distribution of coating and good stain release. To understand physical changes in treated (coated) and untreated cotton fabric samples, tensile strength on Prolific tester and stiffness testing on bending length tester are carried out.

3.3Strength Testing

Table No. 2: Strength testing Observations for Untreated fabric sample

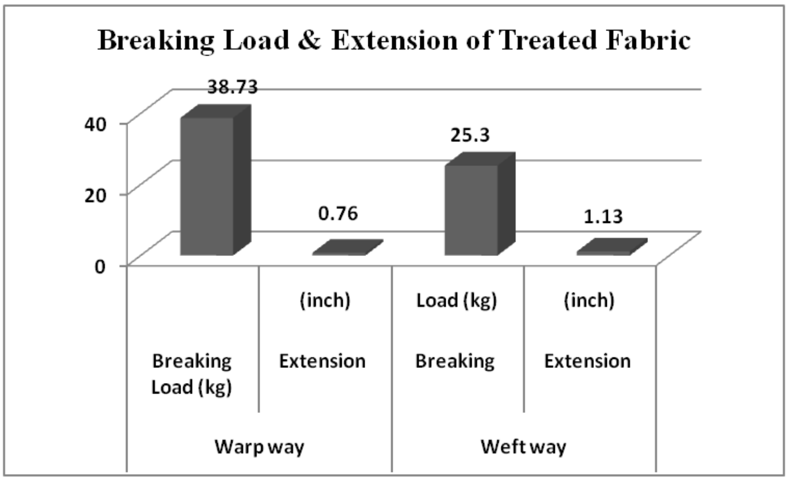
Sr. No.	**Warp way**		**Weft way**	
	Breaking Load (kg)	Extension (inches)	Breaking Load (kg)	Extension (inches)
Avg.	48.06	1.13	28.4	1.66



Graph No.1 Breaking Load & Extension of Untreated Fabric

Table No. 3: Observations for Treated fabric sample

Sr No.	Warp way		Weft way	
	Breaking Load (kg)	Extension (inch)	Breaking Load (kg)	Extension (inch)
Avg	38.73	0.76	25.3	1.13



Graph No.2 Breaking Load & Extension of Treated Fabric

Observation of Table No. 2 & 3 confirms that coated/treated fabric sample show less average tensile strength compared to untreated fabric. It will have bearing on service life of the fabric.

3.4 Stiffness Testing

The Shirley stiffness tester is used. Sample size used is 6” X 1”.

Table No. 4: bending lenth Observations for Untreated fabric samples

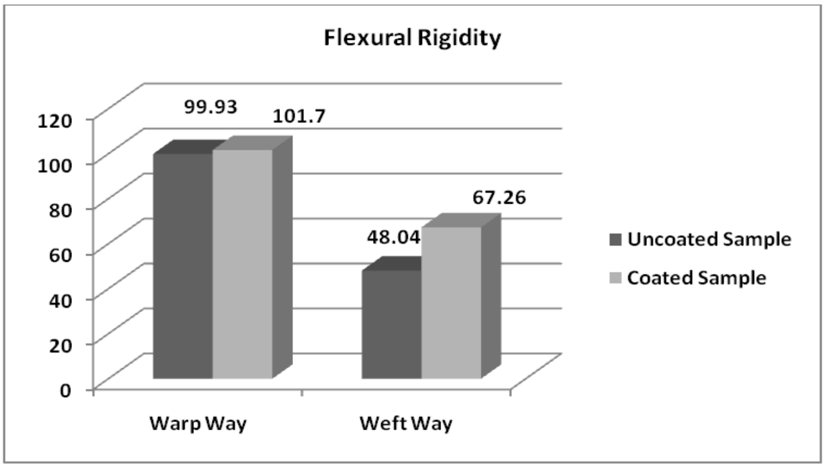
Sr. No.	Warp way				Weft way			
	Face side (cm)		Back side (cm)		Face side (cm)		Back side (cm)	
	Up side	Down side	Up side	Down side	Up side	Down side	Up side	Down side
Avg:	2.66	2.8	2.86	2.86	2.2	2.2	2.3	2.2

Table No. 5: Bending lenth Observations for Treated fabric samples

Sr. No.	Warp way				Weft way			
	Face side (cm)		Back side (cm)		Face side (cm)		Back side (cm)	
	Up side	Down side	Up side	Down side	Up side	Down side	Up side	Down side
Avg :	2.8	2.73	2.73	2.7	2.43	2.43	2.36	2.33

Table No. 6 : Flexural Rigidity (mg*inch)

	Warp Way	Weft Way
Uncoated Sample	99.93	48.04
Coated Sample	101.7	67.26



Graph No.3 Flexural Rigidity of Coated & Uncoated Samples

Examination of Table No 4 & 5 confirms that treated fabric sample average bending length is increased compared to untreated fabric. It can be concluded that TiO_2 treated samples show stiffness compared to original samples. Further, it can be concluded that to make fabric self-cleaning, one has to compromise on durability and draping qualities of the fabric.

IV. Conclusions

Nitric acid hydrolised sol-gel TiO_2 treated fabric samples show good stain loss as compared to acetic acid hydrolised sol-gel TiO_2 formulated in first and third method. Nitric acid hydrolised sol-gel TiO_2 treated fabric samples observed under microscope show uniform distribution coating on treated fabric sample. Physical testing of fabric samples show that one has to compromise on draping qualities and service life of the fabric to make fabric self-cleaning.

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