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Investigation of Sol-Gel Method of Coating Nanoparticles (TiO2) Manufacturing Process by using Design of Experiment

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Abstract:-- **The nanoparticles of Titanium dioxide coatings has good thermal and electrical conductive properties and these nanoparticles are resistant to oxidation, consumption, disintegration, scratch safe and wear in high temperature situations. This property is essential element in the applications of pipelines, castings and automobile industry.The major contribution of the project work is to develop composite materials and enhance the anticorrosion property & increase the life of automobile components materials by using Titanium dioxides nanoparticles were synthesized by using metal alkoxide (Titanium tetraisopropoxide) by sol-gel method. The optimization of process parameter is obtained by using the response surface method (RSM) based on central composite design (CCD).By using Design Expert 9 software find significant values based on ANOVA Table. Based on particle size and antimicrobial activity, the optimized parameters were fixed. Titanium dioxide nanoparticle sol is mixed with paint in different proportions (% v/v) and coating is performed on different metal plates. The performance of coating is tested for smoothness and anticorrosion properties. Last 40 years has major change takes place in coating industry by using new coating technology. Coating gives two main important uses like decoration and protection. In this investigation, the uniform nanoparticle coating of titanium dioxide has been applied on mild steel and Aluminum by using spray coating technique. The coating substrates are placed in salt spray chamber for corrosion test.**

Keywords:-- **Coating, Process optimization, Response surface methodology, Sol-gel method**

I. INTRODUCTION

Corrosion is a broadly experienced issue in the automobile industry. To prevent and keep this issue, industry pioneers frequently run analyses to test the erosion resistance of vehicles. By using simulation, it is the process improved way to deal with reenactment and it saves time, money and resources. The reasons of corrosion in vehicles are formed at the assembling stage or environmental conditions happen on the road. Galvanic corrosion takes place because of the utilization of various metals for different vehicle parts.

In recent years considerable work has been completed on living, non-living mixture materials for enhanced coating properties like thermal stability, corrosion resistance, scratch resistance and abrasion resistance. Coatings provide two essential abilities like improvement and assurance. Around 45% of the coatings material created worldwide are developed to brighten and ensure new development technique.

Generally the coatings are divided into two main markets. The first one is industrial support or defensive coatings connected to oil & gas, petrochemical, paper, water and waste treatment plants and power generation industries. The other one is marine coatings connected to ship business, cargo bearers, tows, voyage boats, yachts etc.

Titanium dioxide is the most significant white paint used in the coatings industry. It is extensively used because it efficiently scatters detectable light, informing lightness, glow and cloudiness when incorporated into a coating. Titanium dioxide is a broadly utilized semiconductor. It is used in various applications like optics, the earth, photovoltaics and sun powered cells, self-cleaning and antimicrobial coatings.

Titanium Dioxide having a tremendous chemical stability, great heat resistance, low electron conductivity and it is helpful for outstanding anticorrosion material. Titanium Dioxide is used as a coating material for manufacturing part of machine design.

Titanium Dioxides have great biocompatibility, excellent antimicrobial activity and tremendous corrosion resistivity

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Fig.1 Corrosion: An Important Concern in Automobiles

The sol-gel process means conversion of a structure from a liquefied sol into a solid gel stage. The advantages of sol-gel method is as follows-

- Colloidal sol easily converts into viscous gels and then quickly converts into solid materials.
- By using sol highly pure and uniform surface can be obtained by using easy process and structure mechanism is possible.
- Sol-gel process is compositional uniformity and the ability to formed equal shape.

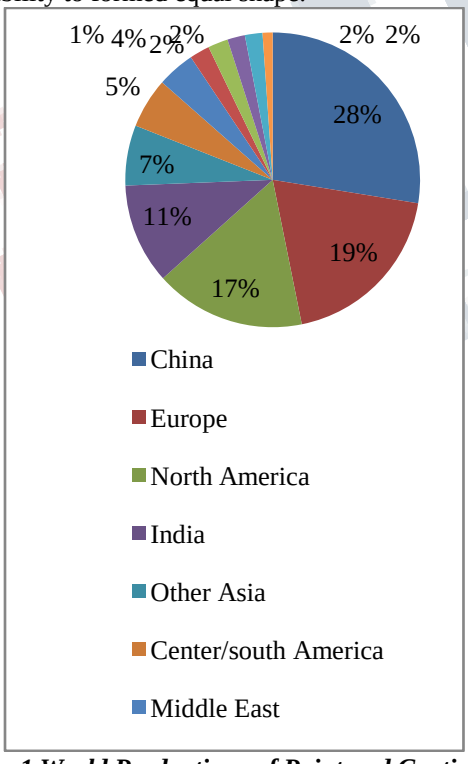

Fig.1 World Productions of Paint and Coatings

Design of Experiment is the statistics and it deals to design the process, to conduct the process, to examine the process and to understand the process to find the factor which is control the value of parameters. This strategic plan and execute the experiment to provide great information of effect of response variable due to one or more factors. By using Design of Experiment improve the product quality by the feedback of customers, to save the time, cost and energy and to improve the process by changing process or material parameter. To develop the mathematical model by using different technique-

- Full Factorial Method
- Taguchi method
- Fractional factorial method
- Multi Regression Analysis
- Partial Factorial Method

II. LITERATURE REVIEW

The uniform coating of titanium dioxide is done without any cracks and improves corrosion resistance of material. Then excellent quality of coating is achieved. By using this sol-gel technique and material is used in pipelines, castings and automobile industry. Sol-gel technique and material is used in pipelines, castings and automobile industry [1]. The sol-gel process form TiO2 which displays photocatalytic movement against some microbes such as Escherichia coli, Pseudomonas aeruginosa & Staphylococcus aureus under visible light. Sol-gel is an alternative technology use to destroy of water contamination [2].The nanotechnology used in manufacture and use of mental, organic and genetic systems. The nanomaterial is used in the maintenance of automobile Industries. [3]. Sometime the nano technique used in production of silica nanoparticles by using low-cost precursor like water glass (sodium silicate). To design the experiments by using response surface method based on the central composite design. By using sol-gel technique obtained 8 nm particle sizes in less costly material and it use for extra application [4].

The developed porous ceramic membranes are formed by using titanium dioxide powder via sol-gel method. Titanium dioxide membrane powder heated at 4500c for 1 hour then stable structure is obtained by sol-gel method.Then this membrane is clean easily and it can be use again. The membrane is made up of simple process and low cost material [5].Titanium dioxides nanoparticles are prepared by sol-gel method under different parameters like TTIP concentration, P-25 loading and gelation pH. The

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process parameters are optimizing by response surface method (RSM) based on CCD Design. By using sol-gel technique form low-price waste-water management [6]. Titanium Dioxide Nano size powder prepared by sol-gel method and XRD phase transformation is obtained. After calcination anatase structure is form.Spherical shaped particles were established through the SEM analysis [7].

By using direct precipitation method equal size of diameter of 20nm ZnO nanoparticles are obtained. By using sol-gel technique Corrosion performance, physical and mechanical material properties of ZPN coating films are calculated. Improvement in the corrosion performance, mechanical properties at low Concentration [8]. To develop anticorrosive siloxane coatings based on Pr6O11–TiO2 composite films for SS 304 substrate by sol gel technique. By using this process improve the anti-corrosion property of substrate [9]. Optimize the process parameters such as annealing temperature, dipping rate and aging effect & without any cracks film is obtained. In this method HAP & TiO2 are coated separately & after that compare their results. The titanium dioxide coating layer gives better surface finish & it is used in medical field [10].The titanium dioxide is use in sol-gel method. The structure of TiO2 is obtained by XRD & FTIR calculate the transmission range of TiO2. The material is useful in the application of cosmetics, sensors; solar cell etc. [11]. Less parameter is obtained by using 33 full factorial experimental designs. The apple pulp is alternative of TiO2 nanoparticle and it is obtained at low cost. [12]

Literature review deals problems of sol-gel method, coating material which is used in mechanical and automobile industries. In this project, coating material is form by using sol-gel method and nanotechnology.

III. METHODOLOGY

A. To develop Experimental set-up-

Titanium dioxide sol is prepared by using sol-gel method. It is very simple preparation method. This process required very simple equipment's and less time. In this process, take round bottom container fitted on magnetic stirrer. Charged distilled water and maintain temperature 60-700c.Once Temperature attend charged acetic acid and maintain temperature 60-70°c for few minutes. Then charged titanium tetraisopropoxide (TTIP) solution with molar ratio H+/Ti, H2O/Ti was added dropwise through syringes under Nitrogen and Maintain reaction mass at 60-70°c for 1 hour. After 1 hour stop the heating and cool the reaction mass at room temperature. The solution transferred in a bottle and kept it at dry place for 7-8 days. After that the solution ready for coating test. This solution is applied on mild steel or aluminum substrate by using spray coating technique.

Fig.3 Experimental set-up

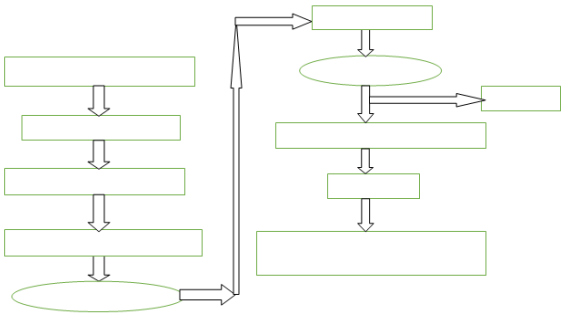

Fig 4 Experimental Procedure

B. Experiment on Titanium Dioxide (TiO2)-

Titanium tetraisopropoxide (TIPP), Glacial acetic acid and distilled water without purification from Cleanchem laboratories LLP, Rabale, Navi Mumbai, India.

The particle size is checked by using Malvern Zetasizer instruments. This test was conducted in the Institute of Chemical Technology, Mumbai. The antimicrobial activity is conducted in Shubhada Research Institute Bio services, Virar, Thane. The antimicrobial activity was conducted by using S.aureus ATCC 6538 microorganism. The spray coating test and salt spray corrosion test is conducted in Nova surface Centre pvt.ltd, Vikhroli.

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C. *To develop Mathematical Model-*

Observing process parameter and responses from experimental set-up. These observations are used to develop the mathematical model by using Design Expert 9. Development on mathematical model by using technique of Design of Experiment. Design of experiment consists of many industrial Engineering Methods. The connection between the response and variables was observed by the following experimental equation in definite form-

- Particle size=201.91+11B+4.65AB-6.55A2-4.85A2B
- Antimicrobial activity = -114.37500+46.26724A +1.65086B-0.48125AB-4.67672A2-2.94181*10-3B2+ 0.05000A2B
  Where, A=H+/Ti
  B=H2O/Ti

D. *To optimize process parameter-*

The developed mathematical models of experiment observations process parameter are to be optimized. The optimizations of variables are required non-traditional optimization technique.

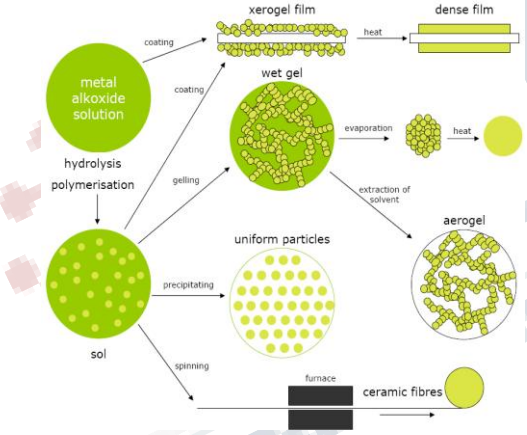

Fig 5 An overview of sol-gel processes (Brinker et al, 1990)

Sol is a stable suspension of colloidal solid particles or polymers in a liquid. Gel porous, three-dimensional, continuous solid network surrounding a continuous liquid phase. A Colloidal (particulate) gels are agglomeration of dense colloidal particles. A polymeric gel is agglomeration of polymeric particles made from sub colloidal units Agglomeration is covalent bonds, van der Walls, hydrogen bonds, and polymeric chain entanglement.

IV. RESULTS AND DISCUSSIONS-

- Design of Experiment Method-Response Surface Methodology (FC-CCD)
- Factors-2
- Responses-2
- No. of levels- 3
- Centre points-5
- No of experiments = 2 3 + 5 = 13
- Software- Design Expert 9

Table 1 Design Data Table

Run No.	Factor 1 H^+/Ti(mol/mol)	Factor 2 H_2O/Ti(mol/mol)	Response 1 Particle Size(nm)	Response 2 (Antimicrobial Test)
1)	5	100	213	19.5
2)	4	100	197.2	19.25
3)	4	120	203.1	16.5
4)	5	100	217.2	21
5)	4	80	200.1	19
6)	5	100	206.4	19.5
7)	5	100	211	20
8)	5	80	194.3	21
9)	6	100	201.8	22
10)	5	120	216.3	17.25
11)	5	100	197.2	20
12)	6	120	214.2	20
13)	6	80	192.6	24

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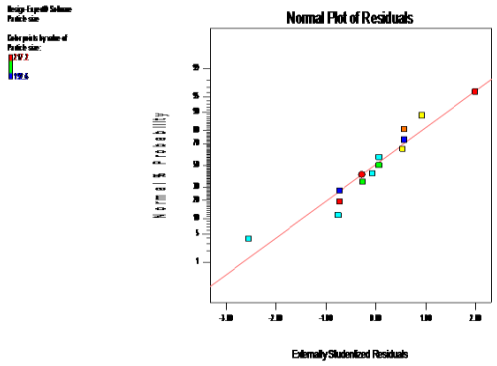
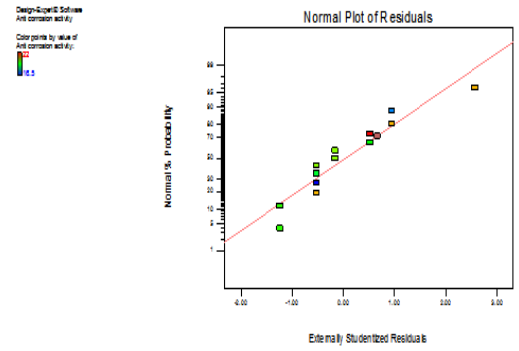
A. Normal Plot of Residuals-

a. Particle size-



Graph 1 Normal Plot of Particle size

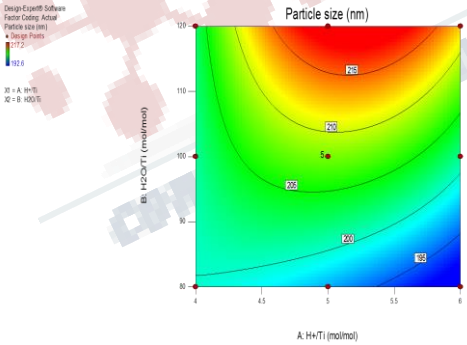
b. Antimicrobial activity-



Graph 2 Normal Plot of Antimicrobial Activity

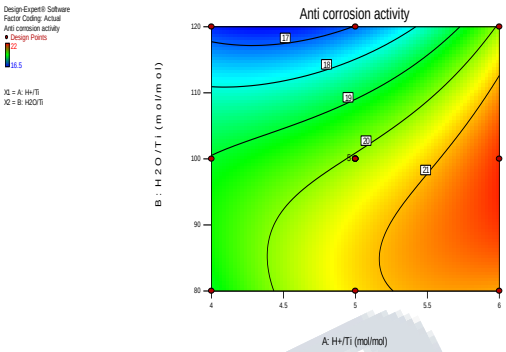
B. Contour Plots-

a) Particle size



Graph 3 Contour Plot of Particle size

b. Antimicrobial Activity



Graph 4 Contour Plot of Antimicrobial Activity

C. Optimization of process parameter- A selection criterion for particle size is less than 200nm and antimicrobial activity is greater than 20.

D. Spray Coating Test-

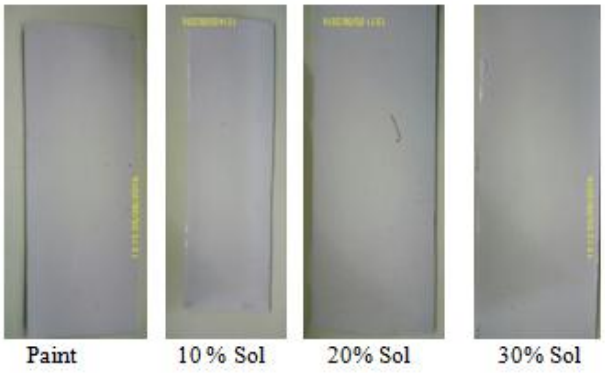


Fig 6 After Coating of Stainless steel Plate

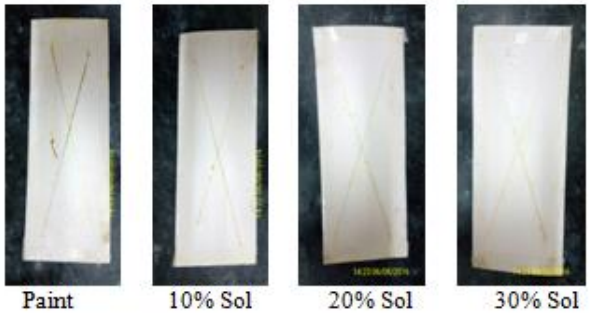


Fig 7 After corrosion Testing of Stainless steel Plates

V. CONCLUSION

By using result and graphs, decide selection criteria for small particle size (<200nm) and higher antimicrobial activity (>20). By using Design Expert 9 software obtained desired parameter. By using these

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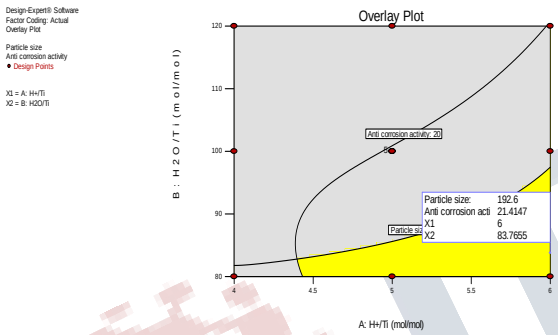
quantities prepared sample for coating in different percentage. Then check the corrosion test by using salt spray chamber on stainless steel substrate.

Table 2 Desired Parameter

Factors	Selection Criteria	Desired Parameter
X1=H^+/Ti	22-16.5	6
X2=H_2O/Ti	217.2-192.6	84



Graph 5 Desired Parameter

VI. FUTURE WORK-

- Similar technique will be useful to prepare nanoparticles which will have applications in the field of fuel cells, batteries, weightless but tough materials for windows and framework of vehicles etc.
- The synthesized Nano composite coating can be extend in natural oil, pharmaceutical reactors to avoid there rusting which will increase their life span.
- By using weightless but tough materials fuel consumption is superior and it increased protection.
- It gives better economics like extensive service life

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