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Efficacy improvement technique of air-filtration unit affected by biofouling using electroless Ni-Cu-P coating

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Moisture from ar is often responsible for biofouling and corrosion on the inner walls of the air filtration system and there by affecting the bacteria colony forming units (CFU Count). This phenomenon is more prevalent in humid environment. Applying electroless Nickel-Copper-Phosphorous (Ni-Cu-P) coating on mild steel helps to prevent bio fouling. An air filtration unit with inner walls coated with nano-TiO_2 and inlet filter mesh made of Luffa and coated activated charcoal and zeolite is used in the experimentation process. The inner chamber is also radiated by band C of Ultraviolet sources (UVC) with 254 nm wavelength. A sudden spike in bacteria colony count has been observed as the air filtration unit remains in off mode for prolonged duration of time. The efficacy of the air filtration unit upon remaining in off- mode for a long time has been determined in the present work considering the bacterial colony count as response. Electroless Ni-Cu-P coating has been applied at selected locations previously affected by biofouling. A predictive model has been developed following response surface methodology using bacterial colony count as response and the composition of the electroless coating bath as the process parameters. Genetic algorithm was applied for the determination of the best possible constitution of chemical coating bath for the minimization of the bacterial colony count. Bacteria Colony formation was significantly affected by the sources of Nickel ion, Copper ion and the concentration of reducing agent in the electroless Ni-Cu-P coating bath. The confirmation run showed that the observed CFU count at the optimum condition converges with the data obtained from Genetic Algorithm.

Keywords: Activated charcoal coating, Air filter, Electroless Ni-Cu-P coating, Natural Luffa filter

Introduction

Air particulate matter (PM) causes different respiratory diseases, including acute respiratory distress, asthma, chronic obstructive pulmonary disease and even lung cancer. These can be reduced using the proper air filtration unit with the provision of arresting PM and bacterial colony formation, bio-fouling. Humid air droplet causes corrosion and fouling in the inner surface of an air filter how due to formation of biofilm adhering to the inner wall of the air filter when it is kept in long condition for a prolonged time. Applying electroless Nickel-Copper-Phosphorous (Ni-Cu-P) coating on mild steel is an economical way to reduce the corrosion and biofouling and there by maintaining a positive air pressure[1]. Electroless Ni-Cu-P coated surface even showed a decrease in the amount of crystallization fouling than in case of uncoated mild steel[2]. Literature shows that the electroless Nickel based coated samples having 11.1% and 13.1% of Phosphorus reduces the rate of corrosion[3]. The addition of different alloying elements affects the corrosion resistance property of electroless Ni-P coating. Studies show that the addition of Molybdenum to Ni-P has little or no effect on the improvement of the corrosion resistance, but addition of Copper, Tin, Tungsten improve the corrosion resistance property of Ni-P coating[4-6]. The surface roughness of the coated inner surface affects the air flow velocity in the air filter. The surface roughness of electroless coating depends on the amount of porosity present on the coated surface, the deposition rate, deposition time, plating condition, addition of surfactant etc.[7-12]. Nucleation takes place when the substrate gets activated before immersing into coating bath. Lower nucleation density of the coating increases the surface roughness of the electroless Ni-P coating[13].

Titanium dioxide (TiO_2) coating irradiated by UVC light and generates negative and positive charges. These charges react with the atmospheric oxygen and water vapour to generates superoxide ion radicals (O_2^{-}) and hydroxyl ion radicals ($OH^{\bullet}$) which further help to abolish the bacterial colonies[14].

Experimental Section

In the present study, the efficacy of an air filtration unit has been improved which has remained in off-mode for a prolonged duration of time. Firstly, the axial intake and radial outlet air filtration unit has been developed with TiO_2 coated inner walls irradiated by UVC lights. The efficiency of the filtration unit has been measured in terms of bacterial colony count. Now the filtration unit was kept inoperative for a period of 7 months. A sudden spike in bacteria colony count was observed when the machine was switched on after 7 months. The same experiment upon coating with electroless Ni-Cu-P at selected locations has shown considerable improvement (decreased bacteria colony count) after the machine was switched on after 7 months. The position which experiences bio-fouling has been identified and mild steel foils coated with electroless Ni-Cu-P coating are pasted with industrial adhesives on those locations. The air filter was tested after a period of 7 months in an environment spiked with *Staphylococcus aureus* Bacteria at Edward Food Research & Analysis Centre Ltd. (EFRAC Laboratories), India. First order regression model has been developed considering a full factorial design with six central points to identify the significant process parameters. A rotatable central composite design of experiment was used to develop a response surface model considering the Bacterial colony count in the test room. The response surface models have been developed to predict the bacterial colony count in that time interval. The effect of the inclusion of electroless Ni-Cu-P coated foils were analysed in this work. The optimum concentration of the elements of the coating bath to minimise the effect of bio-fouling has also been approximated using Genetic Algorithm.

First order Regression model

A first order regression model developed considering a full factorial design of experiment with six additional central points is used to establish a linear relationship between the response and the process parameters. The continuous independent process parameters are represented as x_1, x_2, ..., x_k. The response, y is a random variable. When modelled by a linear function of the independent variables, the approximating function is the 1[st] order model and is represented as Eq. (1).

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \ldots + \beta_k x_k + \varepsilon \quad (1)$$

In Eq. (1) ε is an error component and in Eq. (2) $E(y - \varepsilon)$ is a mathematical expectation of the variable x which gives the predictive model. The fitted first order equation is given in Eq (2).

$$\hat{y} = E(y - \varepsilon) = \hat{\beta}_0 + \hat{\beta}_1 x_1 + \hat{\beta}_2 x_2 + \ldots + \hat{\beta}_k x_k \quad (2)$$

The Student's t-test is conducted on the coefficients of equation (2) to identify the significant process parameters.

Response surface methodology

Response Surface Method (RSM) develops a predictive model using mathematical and statistical tools and techniques to analyze the reliability of the model. To optimise the process parameter subjected to the maximization or minimization of objective function a highly reliable model is needed as an input to Genetic Algorithm (GA). The second order response surface equation shows high reliability. The reliability of the second order model is determined by calculating Fisher's F-value. Response surface is used to approximate the surface around a curvature[15]. A Central Composite Design (CCD) of experiment is used to develop the response surface model. A rotatable CCD consists of factorial points, central points and axial points.

In order to have a curvature in the system, a polynomial of higher degree is used. A second or higher order RSM model is essential to approximate the surface around a curvature. A second-order RSM model can be represented by the Eq. (3)[16]:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_i \sum_j \beta_{ij} x_i x_j + \varepsilon \quad (3)$$

The fitted response surface model is represented in Eq. (4)

$$\hat{y} = \hat{\beta}_0 + \sum_{i=1}^{k} \hat{\beta}_i x_i + \sum_{i=1}^{k} \hat{\beta}_{ii} x_i^2 + \sum_i \sum_j \hat{\beta}_{ij} x_i x_j \quad (4)$$

Genetic algorithm (GA)

Genetic algorithm (GA) is a population-based search algorithm which works on theory of natural selection of evolution[17]. This algorithm is developed based on Darwin's theory of evolution i.e., survival of the fittest. A set of solutions or chromosomes are initialised first as the initial population in zeroth generation. These chromosomes are considered in the form of a randomly chosen string of genes within the ranges of the process parameters. The GA comprises of a selection mechanism, a reproduction mechanism, a crossover mechanism and a mutation mechanism. In selection mechanism, a better fitness function value for minimization or maximization problems is chosen. In reproduction mechanism, the pairing of the chromosomes is done to get new chromosome. The parents are chosen randomly and sorted according to the value of the fitness function to perform crossover. In crossover mechanism, interchanging of the genetic information or genes is done between chromosomes. In mutation mechanism the changing of a particular bit of a chromosome is done to attain the convergence criteria. Finally, the fittest solution survives at the end of the n^{th} generation. This solution optimises the values of the process parameters and the value of the fitness of the objective function at those input values.

Results and Discussion

An axial intake and radial exhaust nano-TiO_2 coated air filtration unit having UV-C lamps housed in the path of air flow was designed in this work[18,19]. After running the system for 20 min, it was observed that the 91.4% bacterial colonies were destroyed due to the reaction of air, water vapour with the superoxide ions and hydroxyl ions which were generated by irradiation of UV-C in presence of nano-TiO_2 coated inner wall of the filtration unit while getting operated in a *Staphylococcus aureus* bacteria filled environment[20]. But maintaining the same level of accuracy was interrupted when the system was kept in non-operative condition for prolonged duration owing to bio-fouling. According to literatures, Nickel-Copper-Phosphorous (Ni-Cu-P) coating has anti-bio-fouling characteristics[1]. As shown in Fig. 1, the corner regions were getting affected by the bio-fouling. To ensure constant efficacy after remaining in shut down condition for prolonged time, electroless Ni-Cu-P coated mild steel foils were attached at the corner regions of the air filtration unit.

The air filtration unit was designed as described in Fig. 2. The inlet and outlet of the air-filtration unit possesses a set of active charcoal and active zeolite coated meshes. The dry Luffa fruits (*Luffa aegyptiaca*) flattened and coated with activated charcoal and activated Zeolite were used as meshes[21]. The CO_x and SO_x are adsorbed by activated charcoal whereas NO_x is adsorbed by activated zeolite[22-25]. The activated charcoal dust and activated zeolite coatings were deposited onto the mesh using methyl cellulose as binding agent. The coating solution containing an aqueous solution of 6% by volume of Methyl cellulose, 10% by volume of Bentonite clay, 5% by volume of activated charcoal powder, 5% by volume of activated zeolite powder were prepared. To maintain the uniform dispersion the solution was stirred at 450 rpm. The coating solution was made ready by heating at 70°C for a period of 50 min and then the solution was sprayed on the sliced Luffa. The coated luffas were kept for 24 h for drying.

The inner surface of the filtration unit was coated with nano-TiO_2 solution (Nano research Laboratory, Jamshedpur) using spray coating process. The inner wall was coated with nano TiO_2 coating[20]. The coating was deposited on mild steel casing by sol-gel technique in the ambient condition using spray coating process with nano-TiO_2 particles.

The electroless Ni-Cu-P coating was deposited on mild steel foils of 0.1 mm thickness having a dimension of 65 mm × 85 mm. The coating condition was optimised in this work to deposit the coating onto the mild steel foils which were attached to the filter walls at the corner portion by industrial epoxy adhesive (Novel Chem, Vadodara, India). To evaluate the optimum condition of the coating deposition, a CCD was followed initially to generate the data

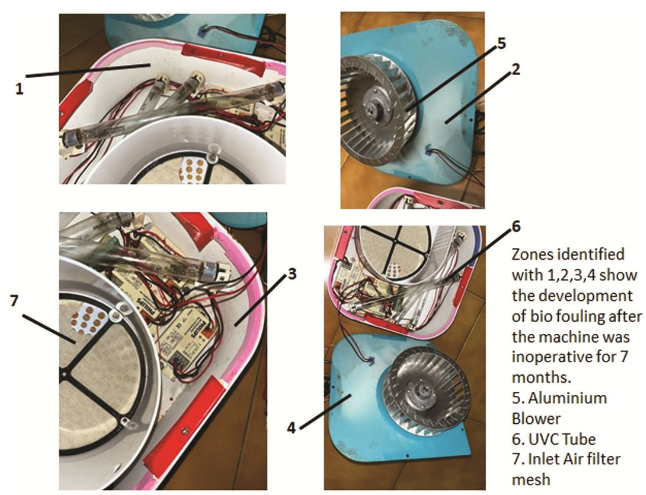

Fig. 1 — Air filtration unit showing the affected regions due to biofouling[19,20,22]

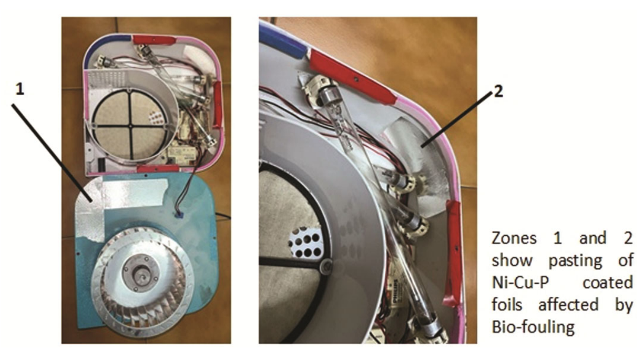

Fig. 2 — Air filtration unit upon attaching the electro less Ni-Cu-P coated mild steel foils

(Table 1). The setup was tested in *Staphylococcus aureus* bacteria filled environment at Edward Food Research and Analysis Centre Limited (EFRAC), Barasat, West Bengal, India. The initial bacterial count was kept at 300 CFU(colony forming units)/m^3. Furthermore, the bacterial colony count was conducted after every experimental run for 20 min which has been noted in Table 2. The first order regression equation and second order response surface model were developed to predict the bacterial colony count with respect to the process parameters. Furthermore, Genetic algorithm was used to approximate the optimal condition to deposit the electroless Ni-Cu-P coating. To deposit the coating on the foils, first the surface was cleaned by emery paper to remove rust. Surface pre-treatment was then performed upon immersing the foils in distilled water, followed by acid pickling, acetone cleaning and rinsing in distilled water to remove the traces of acid and acetone. The deposition reaction has taken place on mild steel foils by catalysis between an anodic solution and a catalytic surface at 85°C. The anodic solution was prepared following Table 1. Before immersing into the coating bath, the casing was dipped into dilute Palladium Chloride solution at 55°C, to catalytically activate the surface to start the auto catalytic reaction. After 1 h of deposition, the foils were taken out. The concentration of the Nickel ion source ($NiSO_4$), Copper ion source ($CuSO_4$) and the reducing agent (NaH_2PO_2) were varied. Ni-Cu-P coated foils after use were tested under Field Emission Scanning Electron Microscope (FESEM) (INSPECT F50, Netherlands).

Preparation of the setup

The air filter was designed following the patents issued to one of the author[18,19,21]. The filtration unit was positioned at a corner of a room having a size of 13.4 ft × 3.4 ft × 8.5 ft. The air filtration unit houses a blower which is driven at high speed by an electric motor. The blower draws in air axially and throws air throughout the casing which was coated with Nano

Table 1 — The conditions to synthesize electroless Ni-Cu-P coating on filter casing

Factors	Role	Range
Nickel sulphate ($NiSO_4$, $7H_2O$)	Source of Nickel ions	23.18 – 56.82 g/L
Copper sulphate ($CuSO_4$, $5H_2O$)	Source of Copper ions	0.86 – 1.54 g/L
Sodium hypophosphite (NaH_2PO_2, H_2O)	Reducing agent	25.908 – 46.092 g/L
Tri-sodium citrate, dihydrate ($Na_3C_6H_5O_7$, $2H_2O$)	Complexant	55 g/L
Sodium acetate (CH_3COONa, $3H_2O$)	Buffering Agent.	20 g/L
Temperature		85°C
pH		5.64
Activation temperature		55°C
Bath volume		200 cm^3
Bath loading		0.15 cm^{-1}
Time		1 h

Table 2 — Observed Air flow velocity of the air filter coated with electroless Ni-Cu-P

	Sl. No.	Concentration of $NiSO_47H_2O$ (g/L) (x_1)	Concentration of $CuSO_45H_2O$ (g/L) (x_2)	Concentration of $NaH_2PO_4H_2O$ (g/L) (x_3)	CFU/m^3 (y)
Factorial points	1	30	1.4	42	236
	2	50	1.4	30	241
	3	50	1	42	231
	4	50	1	30	291
	5	30	1	30	286
	6	30	1.4	30	205
	7	50	1.4	42	221
	8	30	1	42	235
Central points	9	40	1.2	36	239
	10	40	1.2	36	219
	11	40	1.2	36	218
	12	40	1.2	36	240
	13	40	1.2	36	242
	14	40	1.2	36	217
Axial points	15	56.82	1.2	36	254
	16	23.18	1.2	36	215
	17	40	1.54	36	206
	18	40	0.86	36	258
	19	40	1.2	46.092	215
	20	40	1.2	25.91	217

Table 3 — Estimated *t*-values of the input process parameters and their interactions

Response	σ_e^2	σ_β	t_0	t_1	t_2	t_3	t_{12}	t_{23}	t_{13}	t_{123}
CFU count	150.9667	4.344057	366.2475	3.591113	281.9944	7.665645	3.913392	6.952026	0.09208	0.100827

TiO_2 and was irradiated by three UVC lights of 11 W each. The hydroxyl ions released from the Nano TiO_2 photo catalyst destroys the DNA of the bacteria. The air at the outlet of the filtration unit comes in contact with activated zeolite and activated charcoal mesh which adsorbs the gases of oxides of Nitrogen and Carbon. A known volume of air is collected over a liquid culture media and the micro-organisms colony counting was conducted in CFU /m^3 of air.

Statistical analysis and Response surface model

In this present work the bacterial colony count has been considered as the response (*y*) and concentration of Nickel Sulphate (x_1), Copper Sulphate (x_2) and Sodium hypophosphite (x_3) as the input process parameters. The fitted first order regression equation (5) has been developed considering the 8 factorial points and 6 central points (Table 2). The 6 central points have been considered to estimate the error also. The predicted response *i.e.* predicted bacterial colony count ($\hat{y}$) has been depicted in Eq. (5).

$$\hat{y} = 1591 - 15.6x_1 - 1225x_2 - 33.3x_3 + 17x_1x_2 + 30.2x_2x_3 + 0.4x_3x_1 - 0.438x_1x_2x_3 \quad (5)$$

with $R^2 = 81.55$ %

Student's t-test has been followed to find the parameters which are significantly responsible for the variation in response *i.e.*, the bacterial colony count in CFU. The t-values as reported in Table 3 shows that the individual process parameter viz., concentrations of Nickel Sulphate (x_1), Copper Sulphate (x_2), Reducing agent (x_3) and interaction of concentrations of Nickel Sulphate & Copper Sulphate (x_1x_2) and Copper Sulphate & reducing agent (x_2x_3) have significant effect at 5 (number of central points-1) degrees of freedom and 5% level of significance for Bacterial colony count keeping in inoperative condition for seven months.

To consider the curvature effect, a second order response surface equation has been developed considering bacterial colony count as response as shown in Eq. (6).The RSM has been developed considering CCD with 2^3 factorial points, 6 number of axial points and *6* number of central points. To make the design rotatable the axial values has been considered as $(number\ of\ factorial\ points)^{\frac{1}{4}}$ (Ref, 17).

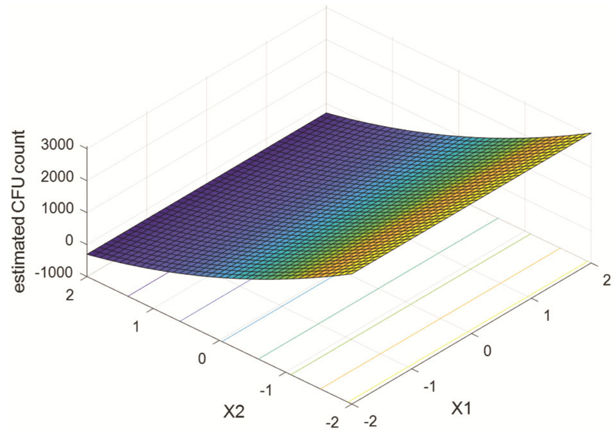

Fig. 3 — Surface and contour plots for $\hat{y} = f(x_1, x_2)$

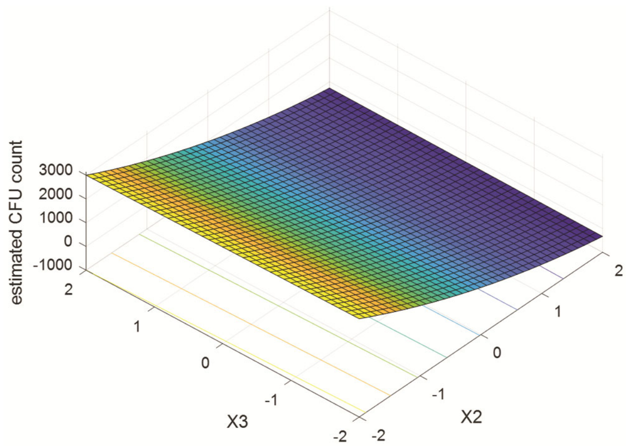

Fig. 4 — Surface and contour plots for $\hat{y} = f(x_2, x_3)$

$$\hat{y} = 965 - 0.55x_1 - 842x_2 - 8.9x_3 + 0.0524x_1^2 + 105x_2^2 - 0.036x_3^2 + 1.25x_1x_2 + 12.71x_2x_3 - 0.125x_3x_1 \quad (6)$$

$with\ R^2 = 76.31\%$

The three-dimensional surface and contour plots (Figs 3-5) have been plotted using the fitted response surface equation in MATLAB R2009b. Here, it can be observed that, the concentration of Nickel ion source, Copper ion source, reducing agent and the interaction of the concentrations of Copper ion source and reducing agent affects the bacterial colony count significantly.

Fisher's Variance Ratio (*F*-Test) is used to test the reliability for predicting the response surface equations. The Fisher's *F*-ratio is obtained as 9.45 which is less than 10.05, represents that the RSM

(Eq. (6)) fits the observed data reliably with 1% level of significance and with upper and lower degrees of freedom as 10 and 5 respectively. The R^2value of the second order equation is closer to 1 to establish that the model is reliable.

Genetic algorithm (GA)

A population of 10D-dimensional solutions $\vec{X}_i(t) = \{x_{i1}(t), x_{i2}(t), \dots x_{iD}(t)\}$ (i.e. *NP*=10) is initialised randomly along with a within the search range $\{\vec{X}_{min}, \vec{X}_{max}\}$. The range of the process parameters has been mentioned in Table 1. Based on the fitness of the objective function i.e. $\frac{1}{equation\ (6)}$ the solutions are arranged in higher to lower value. The offspring solutions ($\vec{X}_i'(t)$) were developed through mutation of the crossover solutions from the parent solutions ($\vec{X}_i(t)$). The fitness values of the objective function were calculated considering the parent and offspring solutions. These fitness values were compared after every iteration to select the fittest solution having highest fitness value. The following steps were followed for 1000 generations(*G_max*=1000):

i. **Initialization**:
Randomly initialize a population of *NP*=10, D-dimensional solutions
$\vec{X}_i(t) = \{x_{i1}(t), x_{i2}(t), \dots x_{iD}(t)\}$
Within the search range $\{\vec{X}_{min}, \vec{X}_{max}\}$,
Where $\vec{X}_{min} = \{x_{min-1}, x_{min-2}, \dots, x_{min-D}\}$
And $\vec{X}_{max} = \{x_{max-1}, x_{max-2}, \dots \dots, x_{max-D}\}$
for $i = [1, NP]$ and $j = [1, D]$ at $t = 0$
i. Evaluate $f(\vec{X}_i(t))$ for $i = [1, NP]$.
ii. for *t*=1: *G_max*=1000

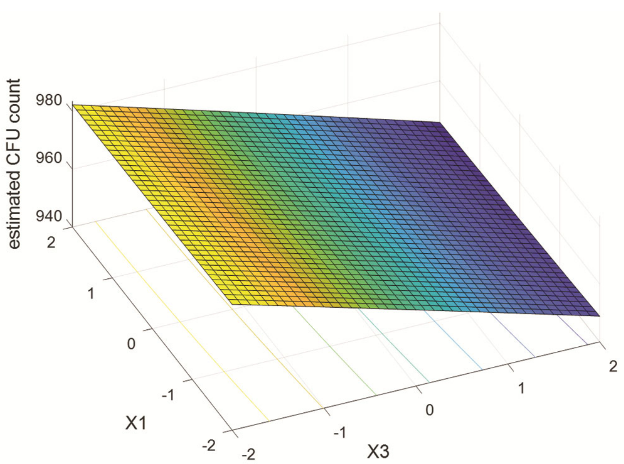

Fig. 5 — Surface and contour plots for $\hat{y} = f(x_3, x_1)$

a) create $\vec{X}_i'(t)$ from $\vec{X}_i(t)$ using

Mutation and Crossover for i =$[\mathbf{1}, \mathbf{NP}]$

b) evaluate $f(\vec{X}_i'(t))$ for$i = [1, NP]$
c) if $f(\vec{X}_i'(t))$ is better than $f(\vec{X}_i(t))$
$\vec{X}_i(t+1)) \leftarrow \vec{X}_i'(t)$.
$f(\vec{X}_i(t+1)) \leftarrow f(\vec{X}_i'(t))$.
d) *t*=*t*+1;
end

A Python code was developed for GA considering the $\frac{1}{\text{response surface model}}$ as the objective function subject to the ranges given in Table 1 of the process parameters, viz., the concentration of Nickel Sulphate, Copper Sulphate and Sodium Hypophosphite. Results of GA code in the 1000th generation yields the minimum *Staphylococcus aureus* bacterial colony count (*y*) of 203 CFU/m^{3}at concentration of Nickel Sulphate (x_1) as 30.13 g/L, concentration of Copper Sulphate (x_2) as 1.54 g/L and concentration of reducing agent i.e., Sodium Hypophosphite (x_3) as 34.87 g/L.

The last 10 generations wise the lowest values of the fitness function along with the corresponding values of the process parameters are shown in Table 4.

Confirmation runs

The coating was deposited in the optimum condition onto mild steel substrate. These electroless Ni-Cu-P coated mild steel foils were attached on identified zones prone to bio-fouling. The filter was used to collect the bacterial colony count in the same *Staphylococcus aureus* environment, which leads to massive reduction in bacterial colony count. It also converges with the result obtained from GA.

Table 4 — Last 10 generations wise values of the1/fitness function along with the corresponding values of the process parameters (Genetic Algorithm Results)

Generation	x_1	x_2	x_3	colony forming units)/m^3
990	30.15	1.54	34.95	*204*
991	30.28	1.5	34.96	*206*
992	30.25	1.53	34.93	*204*
993	30.21	1.49	34.95	206
994	30.22	1.5	34.95	205
995	30.18	1.52	34.93	205
996	30.29	1.51	34.89	205
997	30.28	1.53	34.86	204
998	30.25	1.53	34.84	204
999	30.17	1.54	34.88	203
1000	30.13	1.54	34.87	203

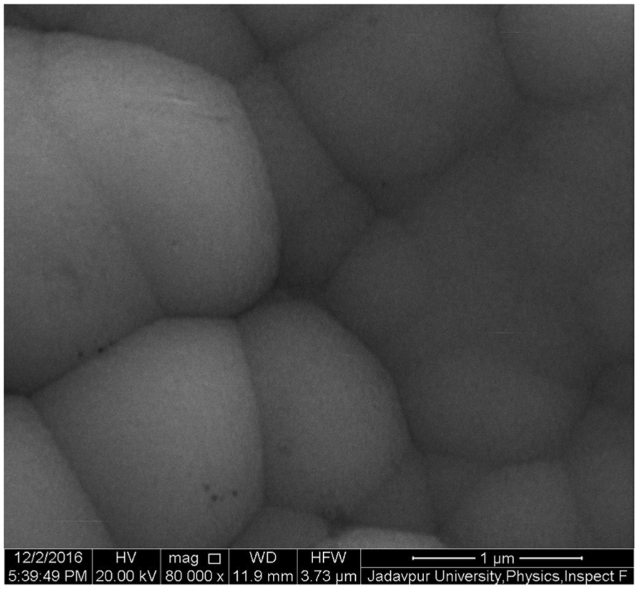

Fig. 6 — The FESEM micrograph of the as-deposited Ni-Cu-P coating deposited in the optimum condition

Characterization of the electroless Ni-Cu-P coating deposited in the optimum condition

The as-deposited Ni-Cu-P coating, deposited in the optimum condition, has interconnected, round, disc shaped grains over the structure with low porosity level as observed from the FESEM micrograph as shown in Fig. 6[26]. It is appeared that presence of low porosity level causes less biofouling or bio corrosion. Occasionally, heaps of particles were observed which was developed due to nucleation and the growth around the nuclei. The coating was deposited overlapping the mild steel plate surface.

Conclusion

The present work was conducted to improve the efficacy of an air filtration unit by solving the problem of bio-fouling. The Student's t-test has reported that the individual process parameter viz., concentrations of Nickel Sulphate (x_1), Copper Sulphate (x_2), Reducing agent (x_3) and interaction of concentrations of Nickel Sulphate & Copper Sulphate (x_1x_2) and Copper Sulphate & reducing agent (x_2x_3) significantly affect the Bacterial colony count (CFU/m^3) during operation after keeping in inoperative condition for seven months. Minimization of bacterial colony count, not violating other process parameters, is obtained by best possible combination of three surface coating parameters using Genetic algorithm. The study would immensely help in designing the process parameter for the development of indoor air filtration system. The minimum *Staphylococcus aureus* bacterial colony count (y) of 203 CFU/m^3 was observed at concentration of 30.13 g/L Nickel Sulphate, concentration of Copper Sulphate 1.54 g/L and concentration of reducing agent as 34.87 g/L within the ranges of the already mentioned parameters of the electroless Ni-Cu-P coating bath.

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