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Prediction of size, precursor ratio and monodispersity of silica nanospheres though adaptive neuro- fuzzy inference system

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ABSTRACT

Highly monodisperse nanostructures are becoming the centre of focus in the field of material science towards the application of sensors, photocatalysis, gas sensing, antibacterial activity, drug delivery and energy applications. Monodisperse resembles uniform nanostructures towards better device performances and applications. In order to minimize the reaction time and characterization costs, an attempt has been made for development of a prediction model for the synthesis of monodisperse silica nanospheres using Adaptive Neuro-Fuzzy Inference System (ANFIS) software. Experimental parameters of the Stober method such as precursor's ratio (ethanol, water and ammonia), Tetraethyl Orthosilicate (TEOS) and sphere size were predicted in the model. The results from the prediction model were used for carrying out experimentation on thin films using vertical deposition technique. The prepared substrates were characterized by FE-SEM & XRD analysis. Obtained Experimental results shows that nanosphere with a size range of 200- 250 nm will form monodisperse layer. XRD analysis confirms the amorphous nature of SiO_2 film. ANFIS has predicted best suitable size of silica nanospheres and optimized precursor's ratios for attaining highly monodisperse structure. The optimized parameters predicted from the ANFIS network matches well with the experimental results.

1. Introduction

Silica nanostructured have been widely used for drug applications, biosensors, bioimaging, Surface Enhanced Raman spectroscopy, gas sensing and acts as template for lithography techniques [1–3]. There is huge demand for low-cost material for advance applications. The different nanostructures of silica are rods, cubes, spheres, wires and pillars. Among the all-spherical particles have been widely used for above applications. Nanosphere has high surface energy because of its surface tension. Nanosphere have small particle size; thus, they are suitable to be administered orally, locally and systematically [4]. Silica Nanospheres have been synthesised using techniques such as sol gel, Coprecipitation, hydrothermal synthesis, Solvothermal method, lithography, chemical reduction and physical deposition techniques. Among these all, Stöber [5,6] method has been widely used by most of the researcher for obtaining desired spherical size by easiest way at low cost. Stöber method is the extension of sol gel method, where the particles undergo hydrolysis and condensation process. The particle size strongly depends on the water and ammonia concentration, but also on the nature of the alcohol used as a solvent. While using alcohols of higher molecular weight, the reaction is slowed down and both median particle size and the spread of the size distribution increased simultaneously. The increase in the size confirms that concentration of these parameters (TEOS, ethanol, ammonia, water) plays an effective role in Stöber reaction and is so complex that each of single parameters directly brings changes in resultant nanoparticles size. After synthesis, it is necessary to bring synthesized silica in working field to use it for different applications and which is only possible if it get deposited onto substrates in wet form. Deposition of silica nanospheres on substrates is carried out by methods such as self-assembly, atomic layer method, spin coating, anisotropic etching, among above spin coating and vertical deposition techniques are simplest and low cost [7]. The deposition process carried out by vertical deposition method helps to get uniform monodispersed structure. In vertical deposition techniques the most important parameters are precursor ration and withdrawing time [8]. Using vertical deposition method SiO_2 nanospheres were deposited on to glass slides for different dipping time. In the present work we have made an attempt for minimizing experimental work using theoretical approach of Adaptive Neuro Fuzzy Inference System (ANFIS). ANFIS is one of the prediction network models which connects the experimental input/output

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Table. 1
Stöber data set of monodisperse silica nanospheres.

S.No	Parameter fixed	Minimum	Maximum
1	Size of silica nanospheres	45nm	700nm
2	TEOS	0.1ul	18ul
3	Ammonia	0.37ml	100ml
4	Water	2.5ml	50ml
5	Ethanol	0.4ml	120ml

and transforms it into a mathematical model for further prediction of results [8].

The main objectives of this work is to 1) Creation of a robust prediction model with Adaptive Neuro-Fuzzy Inference System (ANFIS) to optimize the particle size of silica nanospheres to form monodisperse thin film. 2) Examine the effect of different parameters (TEOS, ethanol, ammonia, water) on particle size of silica nanospheres 3) The predicted precursor ratio were chosen for further experimental validation and it was concluded that 200nm size is the best one for monodisperse coating. The present work will help researchers to fabricate monodispersed nanospheres for various applications. As theoretical simulation well matches with experimental work, the experimental data could be used for device fabrication for various application such as bio sensor, gas sensing and many more. Since there are multiple deposition methods and available nano-structures for various applications, it becomes tedious for the researchers to identify the best selection from the multiple combinations of parameters.

This could save researchers time and avoid laborious calculation and analysis work. The suggested parameters from the present work will save money and helps them to avoid repetitive characterization of samples.

2. Simulation and Theoretical Predication

For optimization of parameters and to maintain the reaction at low cost we need to run simulation. Artificial Neural Network (ANN) and Fuzzy Logic (FL) are the most common of soft computing methods due to their acceptable accuracy within the simulation. In the present work, we have optimized the condition for synthesis of nanospheres using Adaptive Neuro-Fuzzy Inference System (ANFIS) [9]. The system is designed as INPUT, OUTPUT and CPU. Synthesis of nanospheres requires total five parameters such as TEOS, Ethanol, water and ammonia.

2.1. Adaptive Neuro-Fuzzy Inference System (ANFIS)

These parameters were taken as input data and shown in yellow colour. The input data are fed and output results are confirmed as F(u). The white color box indicates that the ANFIS software which works like a CPU. To run the programmes, the following datas were taken from literature survey and experimental work. In the below attached table the following data's are represented as stöber datas. As this whole process needs iteration and minimum fixed parameters, the condition followed as for iterations process.

2.2. Input Data for Size of Silica Nanospheres

The membership function plots the range of the size from minimum to maximum value as shown in Fig. 1 Red color shows low input value, here the value fixed as 45nm.

2.3. Input Data for TEOS

Fig. 2 shows the input data for TEOS. The data is ranging from 0.18ml to 18ml.

2.4. Input Data for Ammonia

The third input data is Ammonia. Ammonia acts as catalyst and plays vital role for synthesis of spheres. The membership function plots the range of the size ranges from 1.2 ml to 100 ml. in Fig. 3

2.5. Input Data for Water

The fourth input data is water. From the Stöber method literature survey the size ranges from 1.2 ml to 53.6 ml in Fig. 4

2.6. Input Data for Ethanol

The fifth input data is ethanol. From the Stöber method literature survey here the size ranges from 0.14 ml to 124 ml.

After fed input data, ANFIS software is running. The first hydration process is in training error which minimizing the error of input values as shown in Fig. 6

From the above five input parameters from maximum to minimum

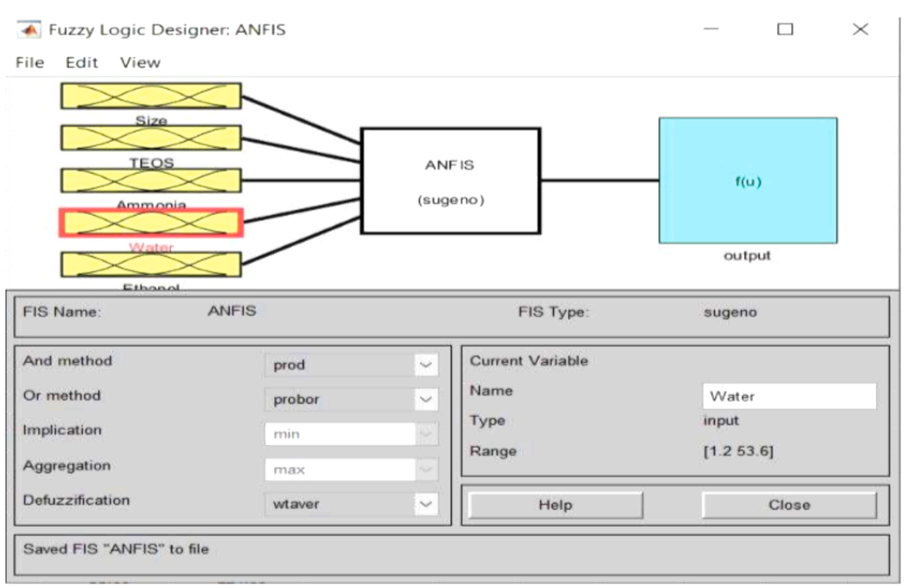

Fig. 1. Theoretical Setup of ANFIS Method

Table. 2
Input data for ANFIS using literature survey and experimental results

S. No.	Size	TEOS (ml)	NH_3 (ml)	H_2O (ml)	Ethanol (ml)	Pore density	Ref
1	520 nm	0.20	2.90	53.60	58.60	Poor	[10]
2	310 nm	0.28	4.30	12.80	40.70	Good	
3	260 nm	0.28	4.30	14.00	40.70	Poor	
4	50 nm	0.20	0.43	53.6	58.60	Very poor	
5	45 nm	0.35	0.50	3.60	1.20	Good	[11]
6	65 nm	0.31	0.51	2.50	15.00	Poor	[12]
7	100nm	5.64	3.00	6.00	38.00	Good	[13]
8	120nm	0.11	25.00	2.29	99.50	Very poor	[14]
9	785nm	0.17	2.00	6.00	50.00	Good	[15]
10	225nm	2.00	5.00	3.00	100.00	Poor	[16]
11	100 nm	0.10	1.10	8.80	13.59	Good	[17]
12	200 nm	0.40	1.20	6.40	13.15	Good	
13	300 nm	0.40	0.56	7.04	13.21	Good	
14	450 nm	4.50	9.00	25.00	62.00	medium	[18]
15	250nm	0.20	0.87	1.20	0.14	Poor	[19]
16	140nm	2.09	3.90	3.08	50.00	Good	[20]
17	700nm	0.67	0.54	4.19	20.00	Good	[21]
18	432nm	100.00	5.00	350.00	30.00	medium	[22]
19	65nm	0.31	0.51	2.50	15.00	medium	[23]
20	555nm	8.50	98.00	18.00	76.00	Poor	[24]
21	500nm	18.00	36.00	50.00	124.00	Poor	[25]
22	330nm	5.26	9.80	10.80	58.20	Poor	[26]
23	120 nm	1.50	3.00	4.00	50.00	Poor	[27]
24	550nm	8.50	98.00	18.00	76.00	Medium	[28]
25	596 nm	0.28	2.00	14.00	11.30	Very good	[29]
26	423 nm	0.28	1.00	14.00	11.70	Very good	
27	281 nm	0.28	0.37	14.00	12.00	Very good	
28	200nm	0.76	4.00	10.00	50.00	Very good	(In Present work)

values. The following output datas correlated with the experimental work. While changing the size of the sphere, TEOS, ammonia, water and ethanol the output results value from 1 to 5. Here 1 to 5 indicates the results as following: 1 is very bad, 2 as fair, 3 as average, 4 as good and 5 as very good.

2.7. Output data for Input size 300nm, TEOS 0.76ml, ammonia 4ml, Ethanol 50ml and water 10ml respectively

The output result is 3.49 which indicates good results for the corresponding input values. The output datas for 60, 100, 200 and 250 nm sizes were found as 1.54, 2.95, 3.29, 3.32 [See supplementary datas Fig S1& S2] Based on the results we have taken 200nm size as the ideal case for the experimental work and further we have run the simulation to find out the optimized precursor ratio values.

2.8. Output data for Input size 200nm, TEOS 0.2ml, ammonia 0.5ml, Ethanol 10 ml and water 10ml respectively

The output results are 3.77 [Fig S3 supplementary] and 3.86 indicates good results for corresponding input values. As per the above simulation the obtained results shows that precursors are directly proportional to the output results. As the value decreases the output increases.

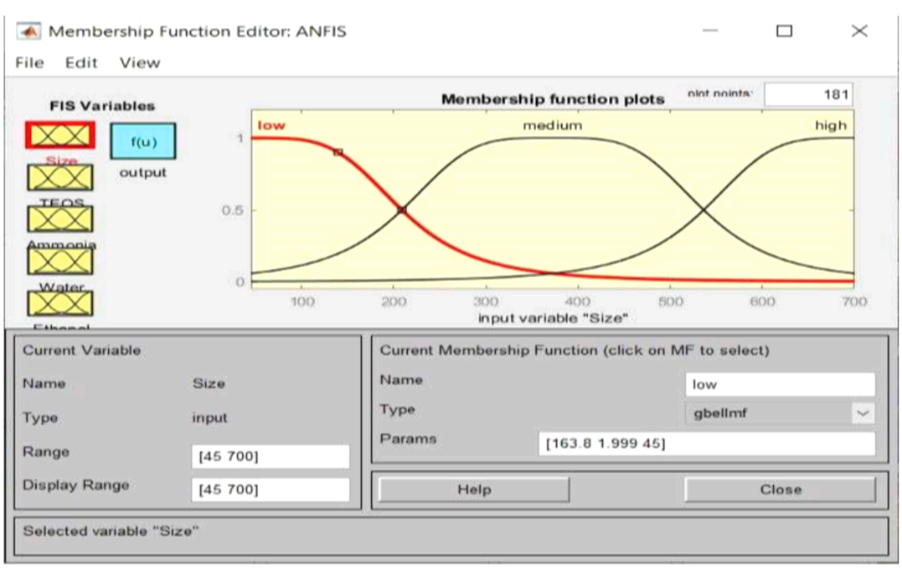

Fig. 2. Input Data of Size

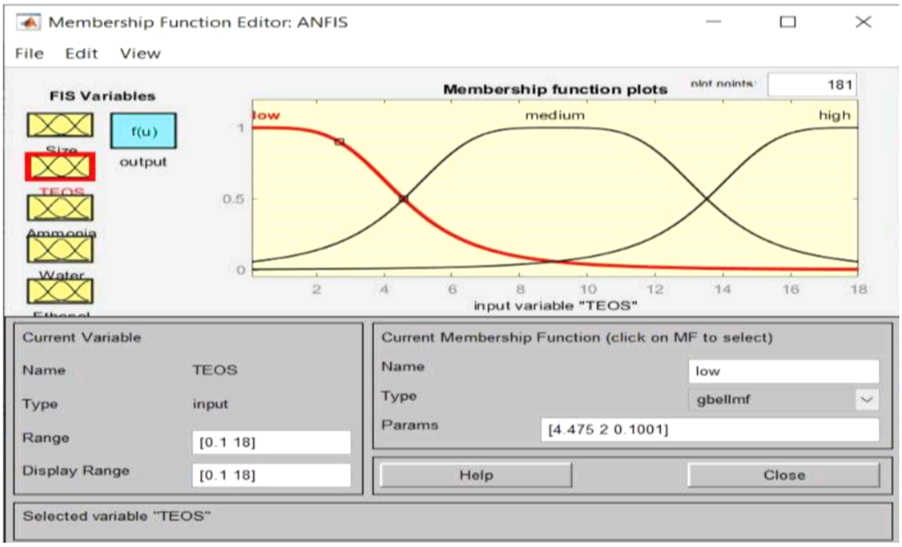

Fig. 3. Input data of TEOS

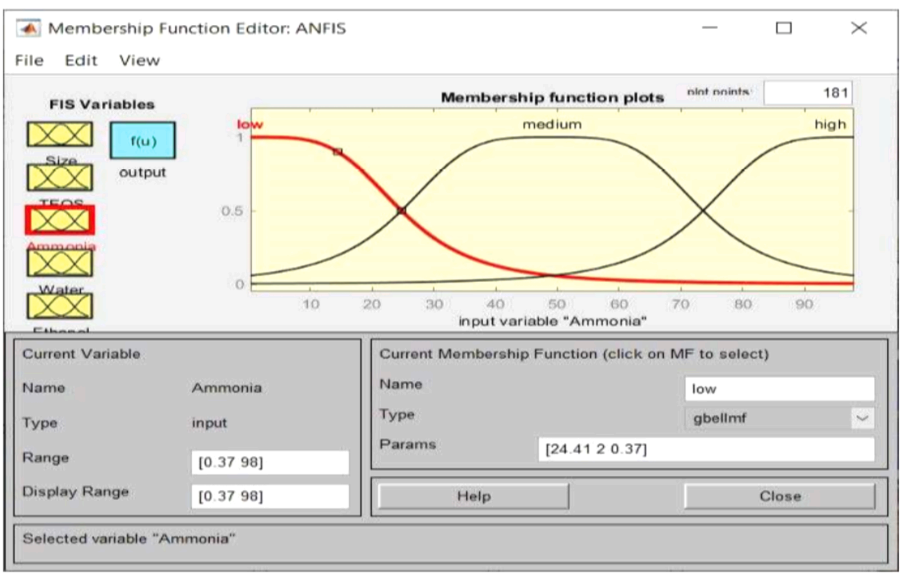

Fig. 4. Input Data of Ammonia

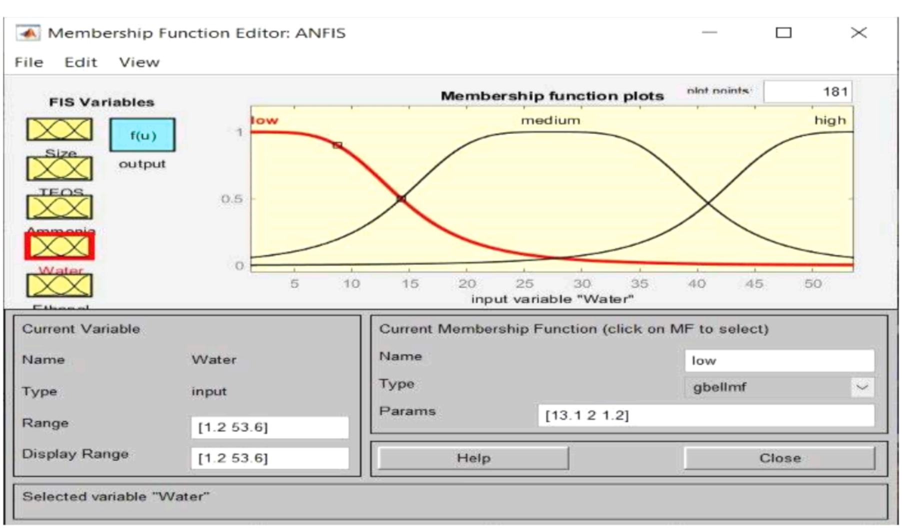

Fig. 5. Input data of water

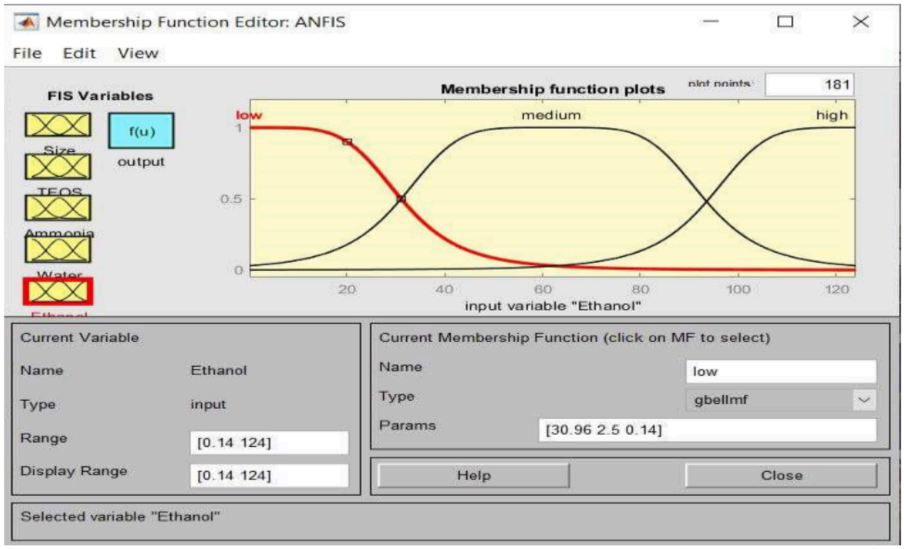

Fig. 6. Input data of ethanol

2.9. Output data for the 200nm size the values changed to TEOS 0.5ml, ammonia 0.5ml, Ethanol 3ml and water 3ml respectively

The outputs results are 4.02 [Fig S4 supplementary], 4.13 indicate good results for corresponding input values. Here the out results are appearing very good as the results are 4.13 the best outcome for all the run profiles.

2.10. Output data for Input size 300nm, TEOS 0.5 ml ammonia 0.5 ml, Ethanol 3ml and water 3 ml respectively

The outputs results are 3.41, 3.77, 3.35, 3.08 indicate good results for corresponding input values. Supplementary Fig S5, and S6 shows that when we change the ratio of TEOS 0.5 ammonia 0.5 ml, Ethanol 3ml and water 3 ml for the size of silica nanospheres 100nm the output will be 3.35 similarly when we can the ratio of TEOS 0.76, 0.28ml ammonia 4,0.37 ml, Ethanol 5, 12 ml and water 10,14 ml respectively the size of silica will be 250 to 280 nm sizes with a output of 3.77.

3. Materials and Methods – Experimental Analysis

3.1. Materials

Ethanol, Ammonia, Tetra Ethyl Ortho Silicate (TEOS) all were purchased from SD fine and glass substrates from the local brands.

3.2. Synthesis of SiO_2 Nanostructure Thin Films

SiO_2 monodispersed nanospheres were synthesized using the stöber method. Silica sol was prepared using tetraethoxyorthosilicate, ethanol, ammonia and de- ionized water. As shown in Fig. 12, 50 ml of ethanol and 10 ml of de -ionized water were mixed and stirred for 10 min in a 250 ml jar. After this, 0.05 ml of TEOS was added to the solution. After 20 min, ammonia was added and the reaction mixture was stirred continuously for an hour. A white colour appears, confirming the formation of silica nanospheres.

The vertical colloidal deposition method is used to synthesize nanoparticle thin films on substrates positioned vertically, relative to the

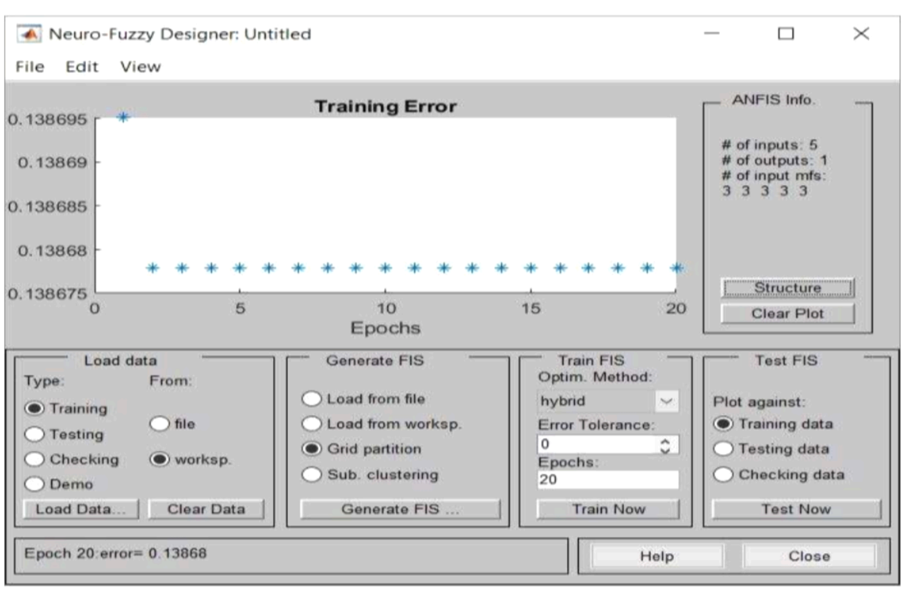

Fig. 7. Training Error

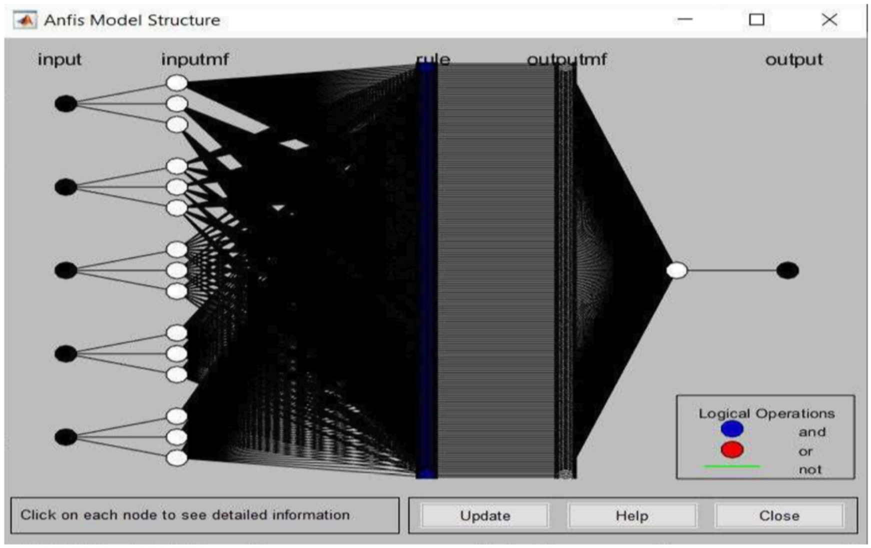

Fig. 8. ANFIS Model Structure

solution surface. As shown schematically in Fig. 13, a microscopic slide was dipped vertically into the solution for different periods of time. After removing from the solution, the slides are dried naturally keeping in vertical position. When the solution evaporates, more substrate is exposed and is coated by the nanoparticle film. The nanoparticles present in the suspension, within the solid-liquid-gas interface, are deposited on the substrate subject to interfacial forces.

3.3. Characterization

Analysis of samples for X-ray diffraction (XRD) patterns was observed on a Panalytical. X'Pert Pro with Cu K_α radiation (1.5406 Å). Field emission scanning electron microscopy (FESEM) studies were performed on a FEI Quanta 200 FEG SEM.Scanning Electron Microscope EVO18 (CARL ZEISS) were used for imaging deposited silica nanospheres. For optimization of parameters and to maintain the reaction at low-cost Adaptive Neuro- Fuzzy Inference System (ANFIS) software is used.

4. Result and Discussion

4.1. XRD analysis

Fig. 14 shows the X-ray powder diffraction pattern of the prepared SiO_2 film. An amorphous peak with the equivalent Bragg angle at $2\theta = 21.8^\circ$ was recorded. The XRD pattern for the SiO2 film has no characteristic peak indicating the amorphous nature of the deposited film.

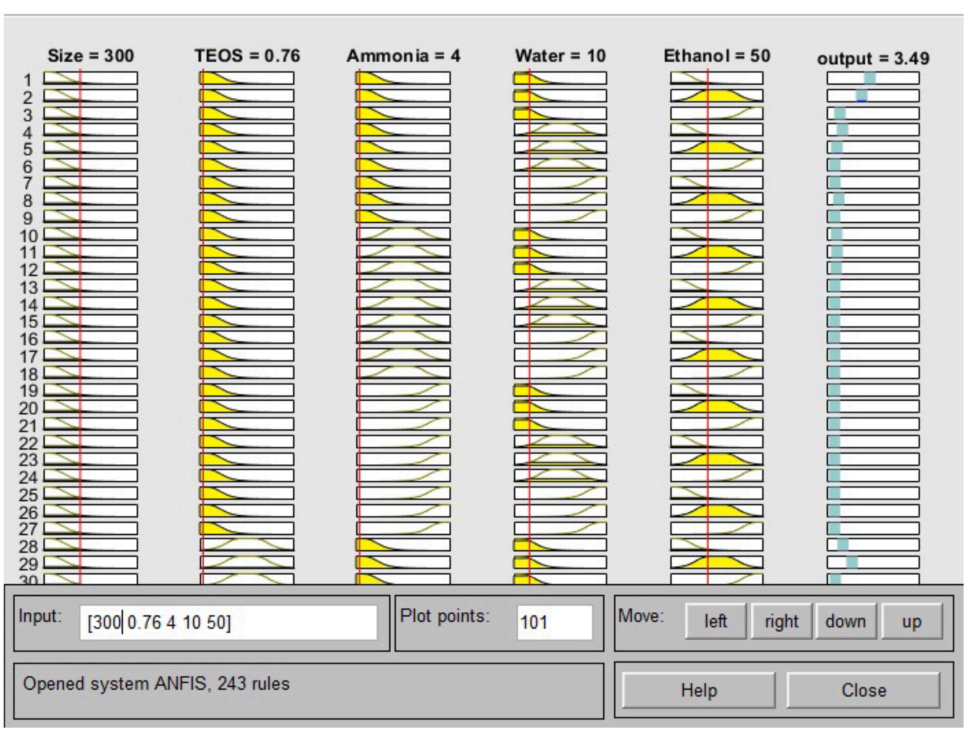

Fig. 9. Output results of ANFIS model

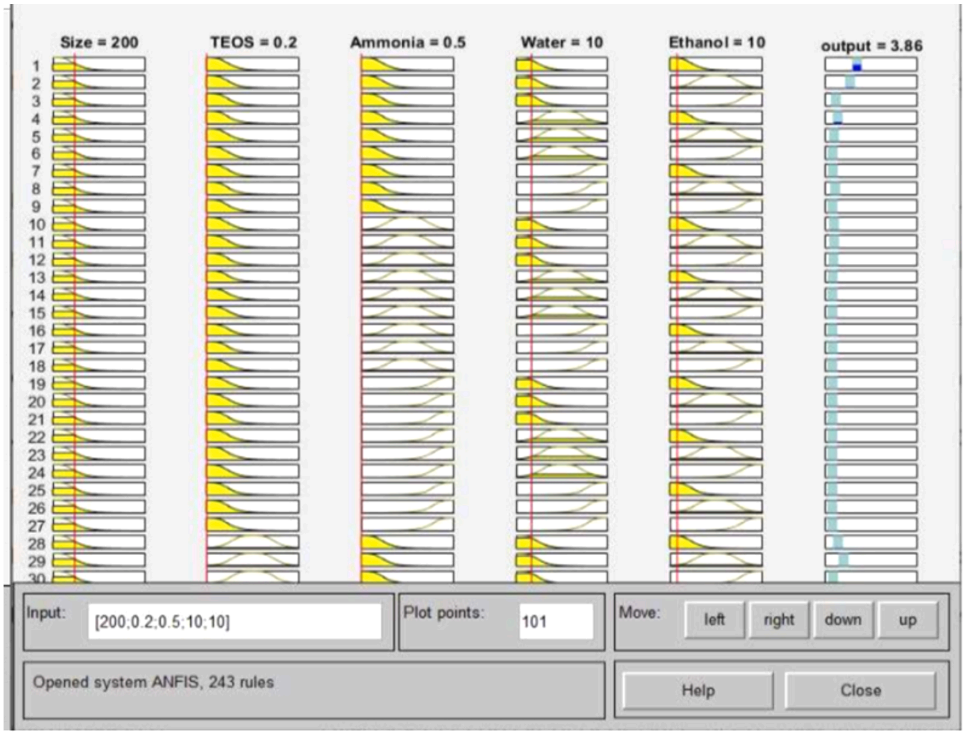

Fig. 10. Output results of ANFIS model

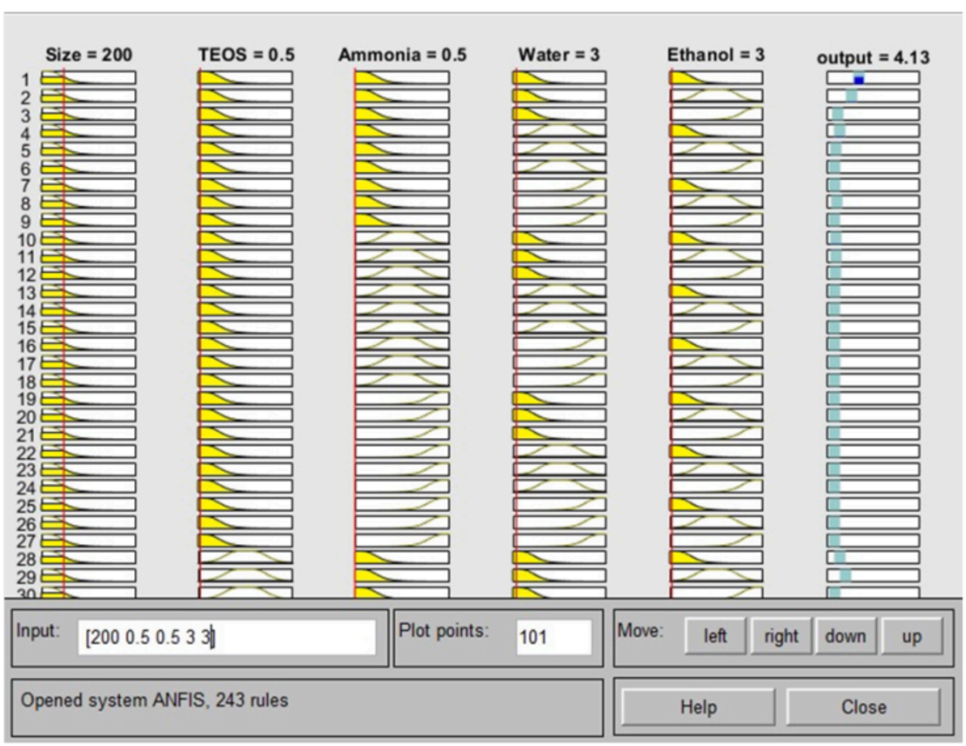

Fig. 11. Output results of ANFIS model

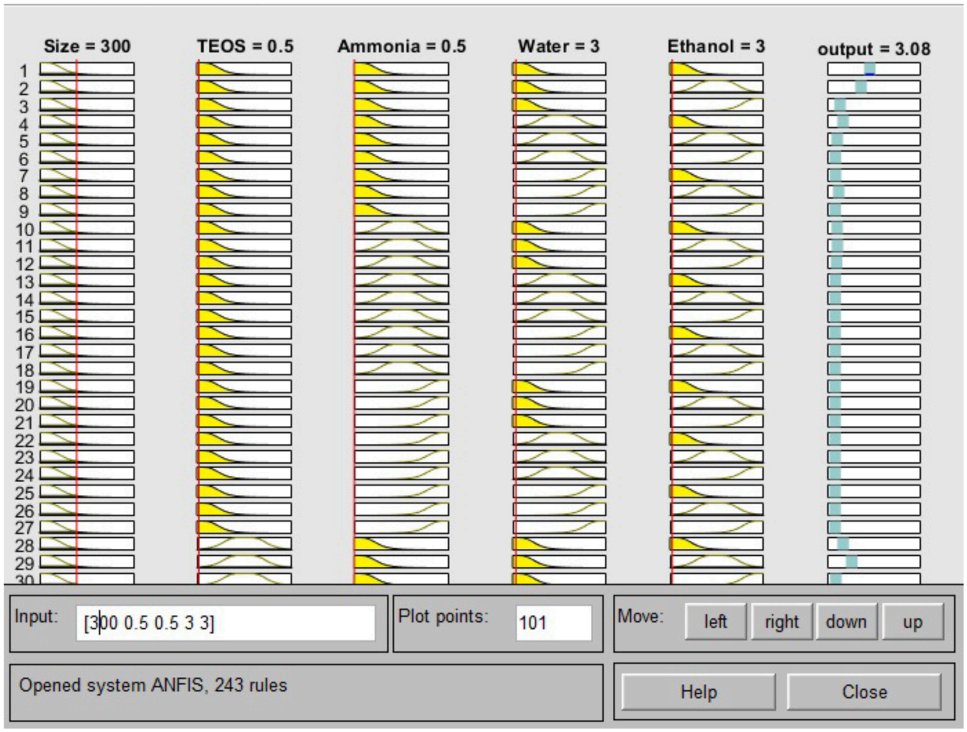

Fig. 12. Output results of ANFIS model

4.2. Morphological analysis

Effects of the precursor ratio and monodispersed: Different precursor ratios were tried before withdrawing the slides from the solution. While changing the ammonia ratio from 4ml to 8ml and TEOS from 0.76 to 1 ul and keeping other aspects same, we found huge variation in formation of silica spheres. Concentration of ammonia did not allow colloidal particles to form a dense monolayer because of kinetic restrictions, and the silica nanospheres were deposited discretely (Fig. 15 (a)). Concentration of TEOS results in the formation of a multilayer structure (Fig. 15 (b)). Typically, the concentration ratio of 1: 8ml, the larger the number of layers of the multilayer structure formed. A lower ammonia concentration and TEOS of 0.76 Fig. 15 (c)) leads to multilayered formation of silica nanospheres with small spheres coming out of the boundary resulting in a packed stack of nanospheres bundle [7].

The surface morphology of synthesized silica nanospheres were characterized using Scanning Electron Microscope. Fig. 15 (d- e) shows the morphology of as deposited silica nanospheres thin films using vertical deposition technique.

On the whole, close-packed monolayer arrays are observed, though spherical stackings and local blanks appear in certain areas, owing to the relative inhomogeneous size distribution of the prepared SiO_2 nanospheres. Defects in the monolayer could arise not only because of impurities in colloids, but also because of the presence of by-products or defective particles. As a rule, small or even missed particles can cause only point defects called ‘mismatches,’ whereas large or twinned particles cause large-scale ‘cracks’ (Fig. 15). In the present work a single monolayer array (Fig. 16) is obtained with ammonia ratio of 4ml, TEOS 0.76 and Water 10 ml and ethanol 50ml. The formation of defects (nanogaps), helps in the applications such as Surface Enhanced Raman spectroscopy [7]. The defects are the sources for the formation of nanogaps, which in turn act as hot spots while coating silver over the SiO_2 layer. The red mark indicates the hexagonal ring like formation throughout the substrate with some gaps, which would further help the silver nanoparticle to fit inside the gaps.

A theoretical result around value 3.29, 3.77, 4.02 and experimental

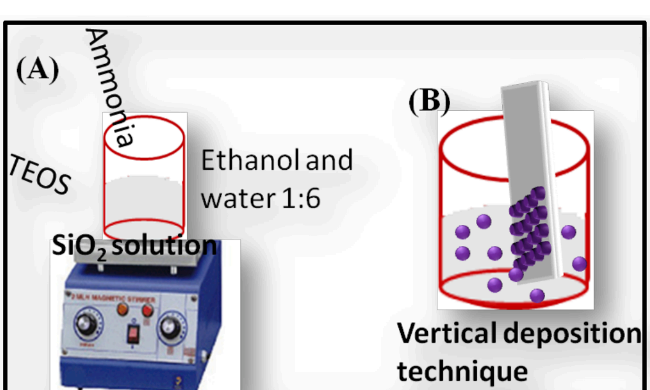

Fig. 13. Schematic diagram for (a) SiO_2 solution preparation (b) vertical deposition technique

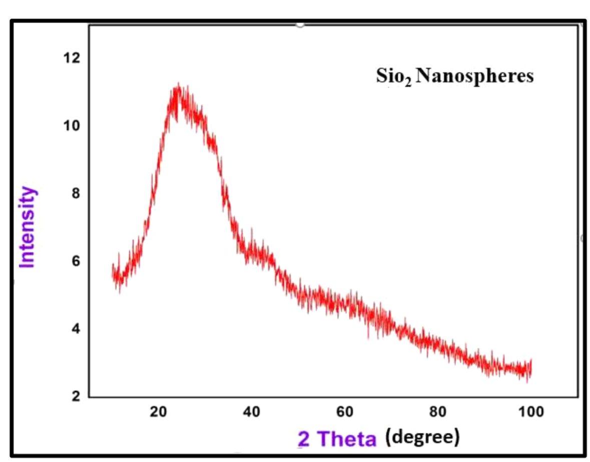

Fig. 14. X-Ray Diffraction Pattern Silica Nanospheres

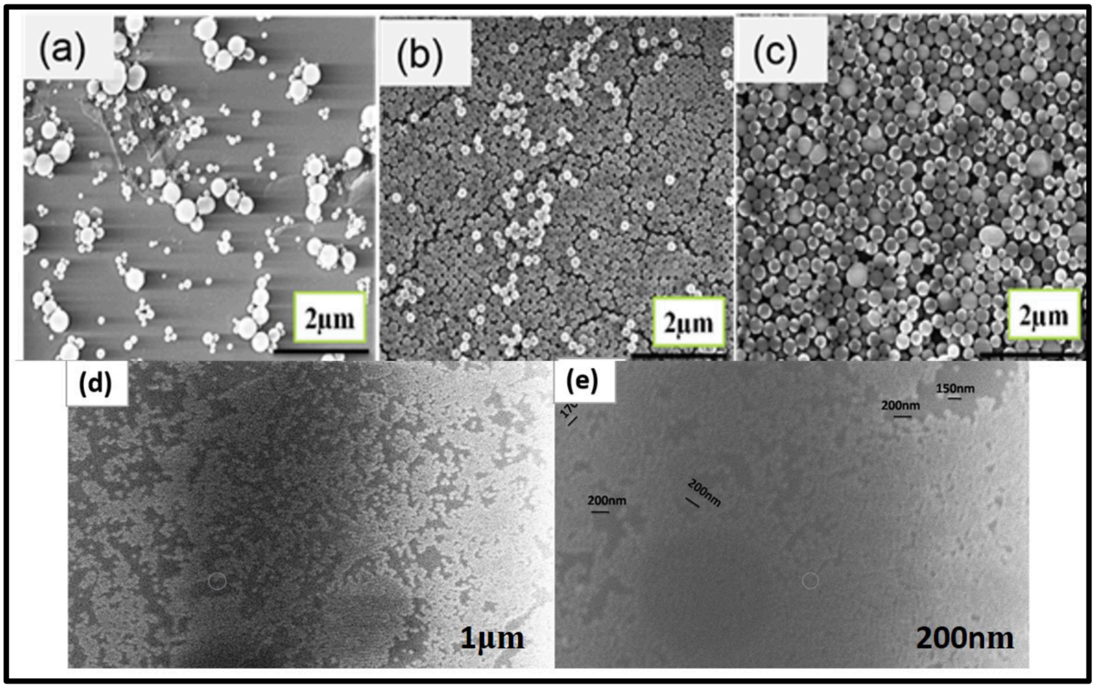

Fig. 15. FE-SEM images of silica nanospheres formed at Ammonia (a) 7ml TEOS 1ul (b) 5ml TEOS 1ul (c) 4ml and 0.76ul SEM images of (d) Silica nanospheres (e) silica nanosphere of different sizes.

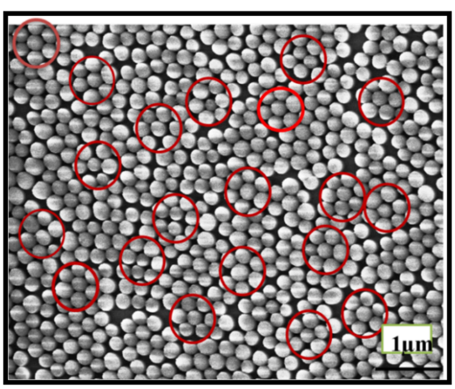

Fig. 16. FESEM micrographs of Monolayer array of SiO_2 nanospheres.

Table. 3
Experimental data vs theoretical values ANFIS parameters

S.No	Size of silica nanospheres	Precursor's value	ANFIS results
1	60 nm	0.76,4,10,50	1.94
	150 nm		2.95
	200 nm		3.29
	250nm		3.32
	300nm		3.49
2	200	1,2,10,5	3.77
3	200	0.2.0.5,10,10	3.86
4	200	1,1,5,5	4.02
5	200	0.5,0.5,3,3	4.13
6	250	0.76,4,10,5	3.41
7	281	0.28,0.37,14,12	3.77
8	100	0.5,0.5,3,3	3.35
9	300	0.5,0.5,3,3	3.08

results value 3.29 for the precursor ratio 0.76, 4, 10, and 50ml for 200 nm well matches.

5. Conclusion

Silica nanospheres have been widely used as a base material for many applications such as drug delivery, bio- sensors, cosmetics, batteries, anti-bacterial agent and etc. Compared to random structure, uniform structures have better conductivity, thermal, mechanical and optical properties. From literature survey, the Stöber method found to be the easiest and low-cost method to prepare silica nanospheres. Uniform size could be achieved only if the number of precursors such as ammonia, water, ethanol and TEOS taken in proper ratio. An attempt has been made to prepare monodisperse silica nanospheres using Stöber method on thin film using vertical deposition technique. XRD results show amorphous nature of SiO2 nanospheres. The SEM results show monolayers of silica having gaps and cracks. While magnification at higher scale the silica nanospheres shows that the individual nanometer size is around 100 nm, for further optimization of parameters, Adaptive Neuro- Fuzzy Inference System (ANFIS) method is used as simulation tool for prediction of sizes. Based on the literature survey data and experimental values results ANFIS simulation has taken 234 rules and output results were analysed from 1 to 5 values. The theoretical result well matches with our experimental results. The results showed that minimum value of TEOS, water and ammonia will result in good uniformity.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.chphi.2021.100042.

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