APPLICATION OF TAGUCHI METHOD TO INVESTIGATE THE EFFECT OF SELECTED PARAMETERS ON CAPACITANCE OF CARBON MATERIALS

Vilas Khairnar

Birla College of Arts, Science and Commerce, Kalyan 421304 (M.S.), India

***Abstract*:** Attempt were made to apply Taguchi method to find out the best experimental condition to get high specific capacitance of synthesized carbon material. Taguchi method involves reducing the variation in a process through robust design of experiments. The overall objective of the method is to produce high product result in minimum steps. From capacitance studies done by cyclic voltammetry technique for carbon materials obtained from plant precursors like Castor seed (Ricinus communis), Ritha seed (Sapindus mukorossi) and Jackfruit seed (Artocarpus heterophyllus) shows that highest specific capacitance is obtained with carbon obtained from Jackfruit seeds. Taguchi optimization methodology predicted that carbonization of Jackfruit seeds at 900ºC temperature for 4 hrs duration and treatment of carbon with 50 % KOH solution gives the carbon of best quality for maximum capacitance. It is also observed that, first major impactful parameter on the specific capacitance is the nature of the precursor and second is treatment with KOH; pyrolyzing time and temperature had very little impact.

***Keywords*:** *Taguchi, Parameter, Carbon, Specific Capacitance, Artocarpus heterophyllus, Seed*

I. INTRODUCTION

With the viability of new carbon electrode materials for supercapacitor, now a days, carbon material synthesized from plant precursors became more popular for its energy application for getting high specific capacitance; but due to large number of precursors, temperature and treatment conditions, process of development in the field of supercapacitor will be time consuming and cost affecting. There was need to reduce the number of experiment without compromising the quality of result. Dr. Taguchi of Nippan Telephones and Telegraph Company, Japan has developed a method based on "orthogonal array" experiments, which gives much reduced "variance" for the experiment with “optimum setting" of control parameter. Thus the marriage of design of experiments with optimization of control parameters to obtain the best result is achieved in the Taguchi method." Orthogonal arrays" (OA) provide a set of well balance (minimum) experiments and Signal to Noise ratios (S/N), which are log function of desired output, serve as objective function for optimization, helping data analysis and prediction of optimum results [1-3].

Taguchi method is a scientifically disciplined mechanism for evaluating and implementing improvements in products, processes, materials, equipment and facilities. These improvements are aim at improving the desired characteristics and simultaneously reducing the number of defects by studying the key variables controlling the process and optimizing the procedures or design to get the best results.

Optimization of quality characteristics using parameter design of the Taguchi method are summarized in various steps by Nian et al., 1999 [4]. Literature survey indicates that there are many attempts to synthesis of carbon material from plant derived and plant based precursors [5-8].

In the present research work, the attempt were made to synthesize porous carbon material from selected plant precursor’s seeds. Different set of experiments were designed using four different parameters like Precursor, Temperature (^{0}C), Time (hour) and Chemical Treatment to synthesized carbon material. The purpose behind this was to get the carbon material which can give the best result of higher specific capacitance. Taguchi method was applied to investigate the effect of selected parameters on capacitance of synthesized carbon materials.

Thus number of variables required to get carbon material which could give the highest capacitance were more than 5-6. In order to carry out minimum number of experiments to get the optimum condition to get carbon material which could give the highest capacitance, Taguchi optimization methodology has been found to be most suitable. Under this statistical technique minimum number of experiments were carried out and got the advantage as if experiments have carried out all possible combinations.+

II. EXPERIMENTAL

To obtain highly porous carbon materials with the aim of getting high specific capacitance, carbonization experimental conditions i.e. parameters were selected. The selected variable parameters were precursors, temperature of pyrolysis, duration of pyrolysis and treatment. To optimize these conditions; Taguchi optimization technique was applied which is based on analysis of Variance (ANOVA) and Analysis of Mean (ANOM) [9-11].

Selection of three plant precursors like Castor seed (Ricinus communis), Ritha seed (Sapindus mukorossi) and Jackfruit seed (Artocarpus heterophyllus) were done and decided to use as precursors for the synthesis of carbon material. L9 orthogonal matrix for these selected plant precursors was prepared [12]. Different parameters and different levels were selected as shown in Tables 1.

Table 1. Different parameters and its levels for L9 orthogonal matrix of selected plant precursors (seeds)

Parameter	Level 1	Level 2	Level 3
A. Precursor	Castor seed	Ritha seed	Jackfruit seed
B. Temperature (°C)	700	800	900
C. Time (hour)	2	3	4
D. Treatment	None	50 % HCl	50 % KOH

For L9 orthogonal array matrix experiments the arrangement of different parameters and levels 1, 2 and 3 are as shown in following Tables 2.

Table 2. L9 orthogonal matrix table for selected plant precursors (seeds)

Exp. No.	Precursor	Temperature (°C)	Time (hrs.)	Treatment
L1	Castor seed	700	2	None
L2	Castor seed	800	3	50 % HCl
L3	Castor seed	900	4	50 % KOH
L4	Ritha seed	700	3	50 % KOH
L5	Ritha seed	800	4	None
L6	Ritha seed	900	2	50 % HCl
L7	Jackfruit seed	700	4	50 % HCl
L8	Jackfruit seed	800	2	50 % KOH
L9	Jackfruit seed	900	3	None

The carbon materials from selected plant precursors (seeds) as mentioned in Table 2 were synthesized using carbonization technique in a single zone furnace [13]. Weighted quantity of plant precursor like (seeds) was taken in quartz boat and kept at centre of single zone furnace. Carrier gas H_2 was allowed to flow into the quartz tube with a fixed flow rate (6ml/min) to make oxygen free atmosphere. After 15 min of flow, furnace was switched on to reach the desired temperature condition. At the end of the desired time the furnace was switched off and allowed to cool at room temperature. Carbon material formed inside the quartz boat was collected. Same way carbon materials from all selected plant precursors were prepared as per above orthogonal array of L9 experiments.

Treatment to synthesized carbon materials were given as per above orthogonal array of L9 experiments. To remove the metal impurities and amorphous nature of carbon from synthesized carbon, it was treated with 50 % HCl. The carbon material was soaked overnight in 50 % HCl solution and washed with distilled water to neural pH condition [14]. For KOH treatment carbon material was mixed with 50 % KOH solution and this mixture was kept for overnight in oven at 80°C temperature. Then after cooling it was filtered and carbon material containing KOH was kept inside the quartz tube of the pyrolysis unit and then heated to 750°C for a period of 1 hour with constant flow of argon. Then after cooling, this mixture was washed several times with distilled water through decantation to remove excess of free KOH present on surface of carbon. Finally it was dried in oven [15].

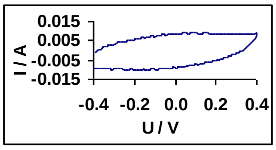
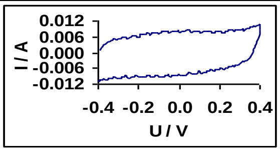
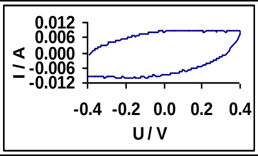
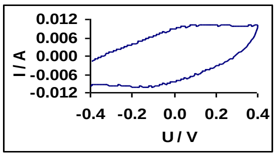
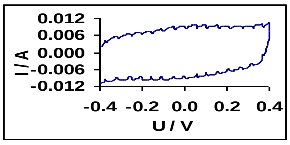
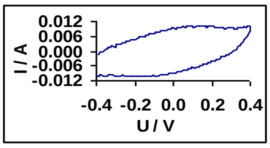
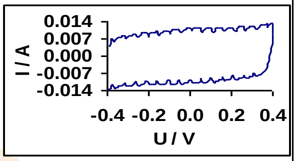
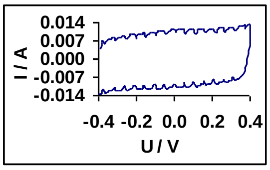
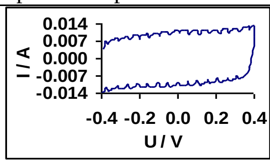
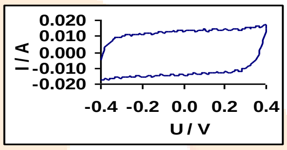
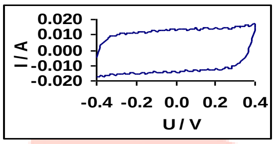
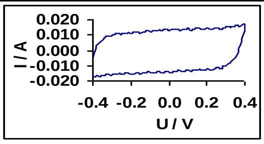
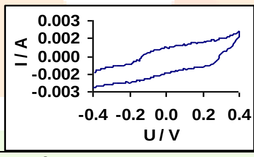
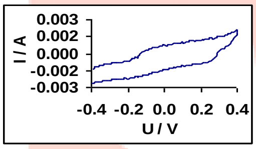
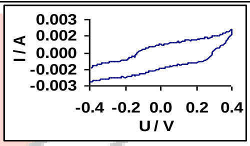
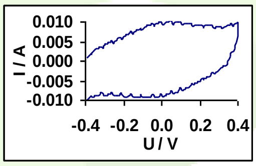
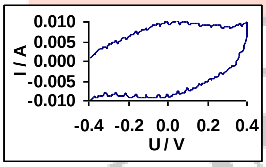
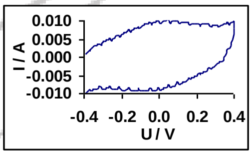
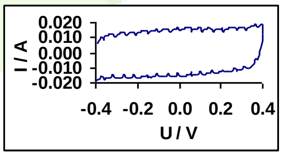
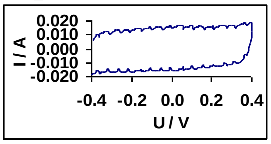
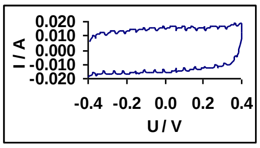
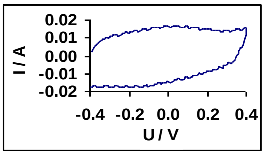
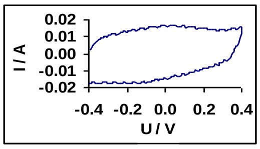
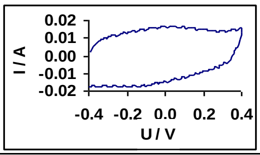
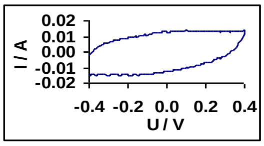
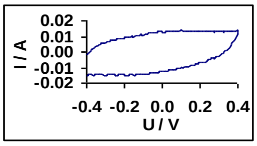
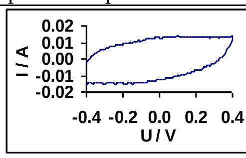
Measurement of capacitance of porous carbon materials synthesized from selected three plant precursors like Castor seed (Ricinus communis), Ritha seed (Sapindus mukorossi) and Jackfruit seed (Artocarpus heterophyllus) was done by two electrode method [16-18]. To find out the potential window, CVs with scan rate using 20mV/s were recorded for all porous carbon materials synthesized from plant precursors. The potential windows were selected in which there were no prominent faradic reaction. Under the selected potential windows, CVs for different synthesized carbons were run at the scan rate of 20mV/s. Specific capacitance based on weight of carbon materials (15 mg / electrode) for each set of experiments were calculated.

III. RESULTS AND DISCUSSION

Measurement of specific capacitance

As per Taguchi method; experiments were repeated three times. Measurement of specific capacitance for all synthesized carbon materials were done by cyclic voltammetry technique (Weight of carbon: 15 mg and Scan rate 20 mV/S) and results obtained are given as E, F and G in the table 3.

Table 3. Cyclic voltammetry curves and corresponding specific capacitance of carbon materials (Weight: 15 mg and CV study at scan rate 20 mV/S)

Exp. No.	Parameters	Cyclic voltammetry curves and corresponding specific capacitance (F/g) of carbon materials		
		E	F	G
L1	A: Precursor - Castor seed B: Temp (ºC) - 700 C: Time (Hrs) - 2 D: Treatment- None	 Specific Capacitance = 40.1	 Specific Capacitance = 39.56	 Specific Capacitance = 40.25
L2	A: Precursor - Castor seed B: Temp (ºC) - 800 C: Time (Hrs) - 3 D: Treatment- 50 % HCl	 Specific Capacitance = 45.76	 Specific Capacitance = 46.34	 Specific Capacitance = 46
L3	A: Precursor - Castor seed B: Temp (ºC) - 900 C: Time (Hrs) - 4 D: Treatment- 50 % KOH	 Specific Capacitance = 53.5	 Specific Capacitance = 51.2	 Secific Capacitance = 54.88
L4	A: Precursor - Ritha seed B: Temp (ºC) - 700 C: Time (Hrs) - 3 D: Treatment- 50 % KOH	 Specific Capacitance = 63.82	 Specific Capacitance = 65.5	 Specific Capacitance = 64.75
L5	A: Precursor - Ritha seed B: Temp (ºC) - 800 C: Time (Hrs) - 4 D: Treatment- None	 Specific Capacitance = 48.9	 Specific Capacitance = 48.73	 Specific Capacitance = 49.6
L6	A: Precursor - Ritha seed B: Temp (ºC) - 900 C: Time (Hrs) - 2 D: Treatment- 50 % HCl	 Specific Capacitance = 55.6	 Specific Capacitance = 56.25	 Specific Capacitance = 55.75
L7	A: Precursor - Jackfruit seed B: Temp (ºC) - 700 C: Time (Hrs) - 4 D: Treatment- 50 % HCl	 Specific Capacitance = 94.63	 Specific Capacitance = 94	 Specific Capacitance = 93.5
L8	A: Precursor - Jackfruit seed B: Temp (ºC) - 800 C: Time (Hrs) - 2 D: Treatment- 50 % KOH	 Specific Capacitance = 101.6	 Specific Capacitance = 102.25	 Specific Capacitance = 104
L9	A: Precursor - Jackfruit seed B: Temp (ºC) - 900 C: Time (Hrs) - 3 D: Treatment- None	 Specific Capacitance = 82	 Specific Capacitance = 83.63	 Specific Capacitance = 82.25

Signal to Noise (S/N) ratio calculation

Taguchi optimization technique is discussed in detail by Siddiquee et al [19]; however a brief description is given here. Basic unit of this methodology is the calculation of 'signal to noise (S/N)' ratio. There are three major ways to use this methodology: (i) 'smaller the better' i.e. when the interest is to find the best parameters to get the minimum amount of the desired product, (ii) 'larger the better' i.e. when interest is to get the largest amount of the desired production (iii) 'nominal the better' i.e. when interest is to get the exact amount of the desired product. Since there was interest in getting the high specific capacitance of carbon, 'larger the better' technique has been used in the present work and the same is discussed here. Since in this research work maximum specific capacitance value is expected, the S/N ratio is calculated using the formula given for 'larger the better' (Eq. 1).

$$S/N\,(dB) = -10 Log_{10}\left(1/n\right)\sum 1/{y_i}^{2} \qquad (1)$$

Where "yi" is the mean response calculated as y =1/n Σyi and n is the number of experiments carried out under similar conditions. All the results obtained after calculation of S/N ratios by using equation 1 are shown in table 4.

Table 4. S/N ratios in orthogonal array of L9 experiments with measured specific capacitance of carbon material

Exp. No.	Precursor	Temp (°C)	Time (Hrs.)	Treatment	Specific Capacitance (F/g)			S/N Ratio
	A	B	C	D	E	F	G	
L1	Castor seed	700	2	None	40.1	39.56	40.25	32.0447
L2	Castor seed	800	3	50 % HCl	45.76	46.34	46	33.2611
L3	Castor seed	900	4	50 % KOH	53.5	51.2	54.88	34.7133
L4	Ritha seed	700	3	50 % KOH	63.82	65.5	64.75	36.2579
L5	Ritha seed	800	4	None	48.9	48.73	49.6	33.8167
L6	Ritha seed	900	2	50 % HCl	55.6	56.25	55.75	34.9427
L7	Jackfruit seed	700	4	50 % HCl	94.63	94	93.5	39.4662
L8	Jackfruit seed	800	2	50 % KOH	101.6	102.25	104	40.2231
L9	Jackfruit seed	900	3	None	82	83.63	82.25	38.3414
					Mean S/N Ratio			35.8963

As discussed in Taguchi optimization technique, arrangement was done for parameters (precursor, temperature, time and treatment) and corresponding S/N ratios for L9 experiment. This arrangement is as shown in table 5.

Table 5. Arrangement of parameters and corresponding S/N ratios.

Exp. No.	Precursor			Temperature (°C)			Time (Hrs.)			Treatment		
	Castor seed	Ritha seed	Jackfruit seed	700	800	900	2.00	3.00	4.00	None	50 % HCl	50 % KOH
	S/N	S/N	S/N	S/N	S/N	S/N	S/N	S/N	S/N	S/N	S/N	S/N
L1	32.0447			32.0447			32.0447			32.0447		
L2	33.2611				33.2611			33.2611			33.2611	
L3	34.7133					34.7133			34.7133			34.7133
L4		36.2579		36.2579				36.2579				36.2579
L5		33.8167			33.8167				33.8167	33.8167		
L6		34.9427				34.9427	34.9427				34.9427	
L7			39.4662	39.4662					39.4662		39.4662	
L8			40.2231		40.2231		40.2231					40.2231
L9			38.3414			38.3414		38.3414		38.3414		
y	100.02	105.02	118.03	107.77	107.30	108.00	107.21	107.86	108.00	104.20	107.67	111.19
y-y mean	-7.67	-2.67	10.34	0.08	-0.39	0.31	-0.48	0.17	0.31	-3.49	-0.02	3.51

Study of Effect of each parameter

Effect of each parameter level (m_i) was determined by using equation 2, in which average value of S/N ratios are calculated for each parameters using analysis of mean (ANOM). For this calculation, the S/N ratios of each experiment with corresponding parameter levels are employed.

$$m_i = \left(1/N_i\right)\sum S/N \qquad (2)$$

Where, *Ni* is the number of experiments conducted with the same parameter levels. Two types of values for S/N ratio are calculated; one is overall mean S/N ratio calculated from the entire experiments *viz.* from 9 experiment of L9 orthogonal array. The second value for S/N ratio is calculated by summation of S/N ratio of each parameter. When this summed value of S/N ratio of each parameter is lower than the mean S/N ratio, then that parameter is to be considered as least effective parameters as compared to those parameters whose summed value of S/N ratio of each parameter are larger than the mean S/N ratio.

The parameters effects, i.e. the contribution of each experimental parameter to the quality characteristic are calculated by the analysis of variance (ANOVA) [20]. This is done by summing the squares (SoS) of variance of all levels of a given parameters which then is normalized with respect to the degrees of freedom (DoF) of the corresponding process parameters (DoF = Number of parameter levels-1). The relations that are used to determine the sum of the squares and the factor effect are given by:

$$\text{Sum of Squares} = \sum_{i=l}^{i=j} Ni(mi - \langle mi \rangle)^2 \quad (3)$$

Where m_i gives the average of the levels contributions for each parameters levels to S/N ratio, $\langle m_i \rangle$ is the average of m_i for a given parameter and the coefficient and N_i represents the number of times the experiment is conducted with the same factor level. In the L9 for first parameter N_i is 3. The factor level of each parameter, is obtained using equation, Ni (mi-<mi>) ^{2}. Sum of squares (SoS) of variances for all levels for a given parameter are obtained using equation 3. This term is divided by degree of freedom (DoF) of corresponding process parameter to obtain factor of effects (FoE) of various experimental parameters (equation 4).

$$\text{Factor of effect (FoE)} = \frac{SoS}{DoF \times \sum (SoS / DoF)} \quad (4)$$

Thus, a plot of S/N ratios for each parameter for its each level is plotted verses the each levels of each parameters. That value of S/N which is greater than the mean values of S/N is considered to be the desired level of each parameter.

Taguchi plot for L9 orthogonal matrix

A plot was made between the S/N ratios for each level of each parameter versus the various parameters (Figure 1.) for carbon materials synthesized by carbonization of selected plant precursors like Castor seed (Ricinus communis), Ritha seed (Sapindus mukorossi) and Jackfruit seed (Artocarpus heterophyllus). This plot suggests that the best conditions for getting the higher specific capacitance are when precursor like Jackfruit seed is carbonized at 900°C temperature for 4 hrs and its carbon formed is treated with 50% KOH solution.

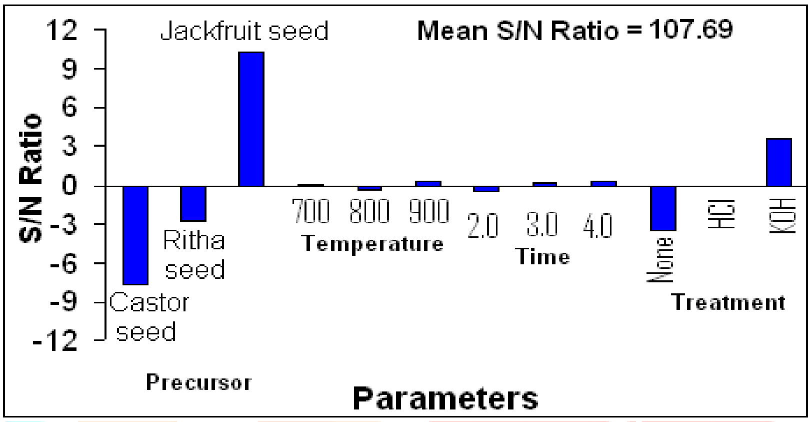


Figure 1. S/N ratio vs. Parameters (considering mean S/N ratio 107.69 = 0; values which are in positive direction are considered as parameters influencing the carbon materials to get the best result)

Amongst the various parameters influencing the results of the specific capacitance, which of them influences the most can be calculated from the Taguchi optimization methodology. Calculations for the values of m_i, <mi>, mi-<mi>, (mi-<mi>) ^{2}, Ni (mi-<mi>) ^{2}, SoS, SoS/DoF, FoE were done as per formulae and finally, percentage parameter effect was calculated using equation 5.

$$\text{Parameter effect \%} = 100 \times \text{FoE} \quad (5)$$

The values of m_i, <mi>, mi-<mi>, (mi-<mi>) ^{2}, Ni (mi-<mi>) ^{2}, SoS, SoS/DoF, FoE and percentage impact of each parameter on specific capacitance is shown in table 6.

Table 6. Effect of control parameters on specific capacitance of carbon materials

Control parameters	Levels	m_i(dB)	SoS	SoS/DoF	Parameter effect (%)
Precursor	Castor seed	33.3397	57.6393	28.8196	87.35
	Ritha seed	35.0058			
	Jack fruit seed	39.3436			
Substrate temperature	700^{0}C	35.9229	0.0840	0.042	0.13
	800^{0}C	35.767			
	900^{0}C	35.9992			
Pyrolysis time in hours	2	35.7368	0.1176	0.0588	0.18
	3	35.9535			
	4	35.9988			
Treatment to the carbon	None	34.7343	8.1468	4.0734	12.34
	50 % HCl	35.89			
	50 % KOH	37.0648			

Taguchi optimization calculations suggest that; capacitance values obtained from these carbons are influenced as 87.35 % on the type of precursor used, 0.13 % on the temperature used for carbonization, 0.18 % on the carbonization time and 12.34 % on the treatment to carbon material synthesized from selected plant precursors (seeds).

IV. CONCLUSION

Specific capacitance studies for carbon materials obtained from selected plant precursors like Castor seed (Ricinus communis), Ritha seed (Sapindus mukorossi) and Jackfruit seed (Artocarpus heterophyllus) shows that highest specific capacitance is obtained with carbon obtained from Jackfruit seeds. Application of Taguchi method to investigate the effect of selected parameters on specific capacitance of synthesized carbon materials predicted that carbonization of Jackfruit seed at 900^oC temperature for 4 hrs duration and treatment of carbon with 50 % KOH solution gave the carbon of best quality for maximum capacitance. It is also observed that, first major impactful parameter on the specific capacitance is the nature of the precursor and second is treatment with KOH; pyrolyzing time and temperature had very little impact. It also concluded that Taguchi method will be more useful in future development of low cost supercapacitor.

V. ACKNOWLEDGEMENT

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