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Original Research Article

Application of Design of Experiment (DoE) for Optimization of Multiple Parameter Resource Constrain Process: Taguchi-Based Fractional Factorial Approach

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ABSTRACT

Traditionally, for process optimization, a single factor is varied single time while keeping all others constant which is not only time and resource-consuming but also scientifically not valid. Generally, the objective is to minimize costs and maximize performance, productivity, and efficiency. Statistics-based solutions may provide better results by utilizing comparatively lesser resources, energy and labour. One such approach is the design of the experiment (DoE). Generally, the organic process includes more than one controlled or input parameter. Therefore, Taguchi's orthogonal design of the experiment may provide a better insight into such a process. Being orthogonal, it provides the impact of each controlling factor on the output characteristic. Therefore, keeping in view, the importance of the fractional factorial DoE, especially for resource-constrained projects, here in the present study a detailed step-by-step approach is discussed by taking an experimental study on the quaternization process of guar gum as a model case.

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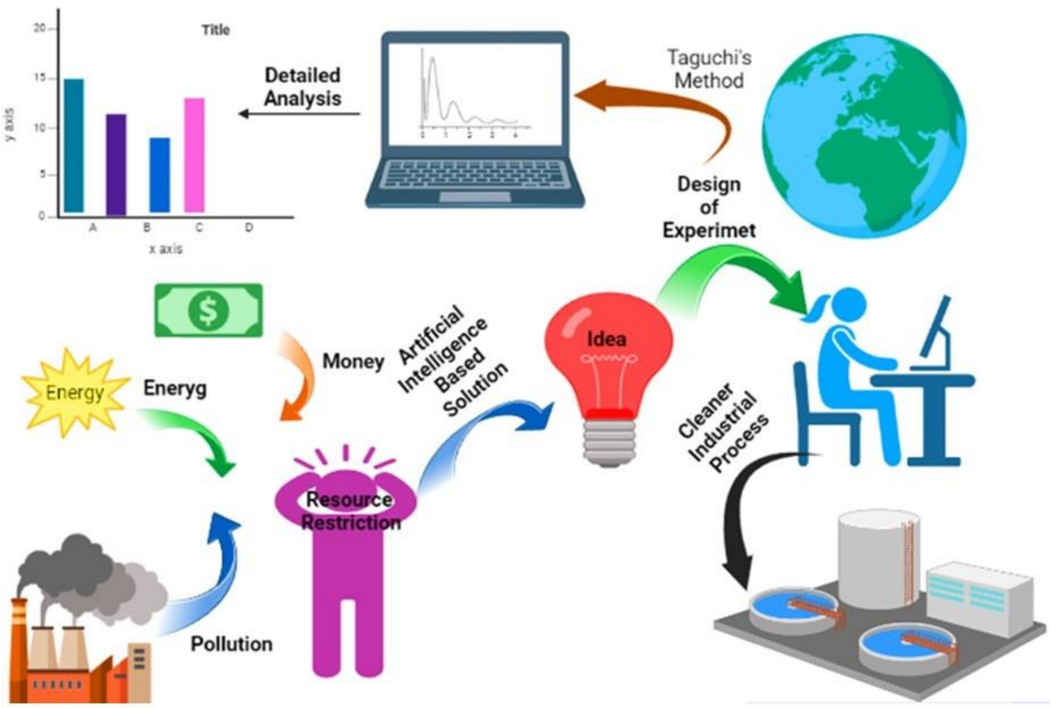
GRAPHICAL ABSTRACT



Introduction

The output of the process is reported to be dependent on several factors. For example, for a chemical reaction, the output of the reaction is significantly determined by the concentration of the reactants, reaction time, reaction temperature, etc. Therefore, to optimize a chemical process there is a requirement for conducting a large number of experiments for obtaining the best reaction conditions for achieving the targeted output.

Optimization involves the application of mathematical tools to find the best appropriate solution for the selected problem from several available alternatives [1]. Recently newer methos are gaining attention in the field of chemistry related research [2-5]. for Generally, the objective is to minimize costs and maximize performance, productivity, and efficiency. The optimization targets may be different for different processes and optimization refers to acquiring the best output. For process optimization, the first and foremost requirement is to identify and select the limits of the controlled parameters/inputs which significantly affect the output or targeted property. The selected factors are varied under their specified limits to conduct the experiments by using various combinations of the input variables [2].

Traditionally, for optimization, a single factor is varied at a time while keeping all others constant. This conventional approach of *one-factor-at-a-time* (OFAT) for optimization encounters many drawbacks [6]. Firstly, it rarely uncovers the optimal conditions as the outcome are highly dependent on the starting point. Nonetheless, it can serve the purpose of coarse estimation of the optimum levels [7-9]. OFAT approach is unable to separate the “noise” (the inherent run-to-run variation of a system) of a process from actual improvement unless a significant number of reactions are repeated using the same conditions.

The scientific approach inherent in the design of the experiment (DoE) eliminates the researcher bias and often will lead to experimental conditions that one had not applied previously [10]. Additionally, DoE provides quick detection of the interactions between factors and their influence on product yield and quality.

Hence statistical method plays an important role in quality improvement efforts [11]. Design of experiment is a methodology for applying statistical analysis during the planning stages of the process rather than at the end of experimentation which enables to build of quality into the product [12]. For process optimization, the most frequently applied DoE are central Box-Behnken [13-17], Central Composite Design (CCD) [18-20] and Taguchi's design of experiments [21-23]. Box-Behnken and central composite are response surface designs that explore the relationships between the controlled variables and one or more response variables. Taguchi is a fractional factorial methodology for process optimization. Fractional factorial designs significantly reduce the number of experiments without compromising the number of process parameters and respective levels.

Generally, the process includes more than one controlled or input parameter. Therefore, Taguchi's orthogonal designs may provide a better insight into the such process while consuming lesser time, labor and resource. Being orthogonal, it provides the impact of each controlling factor on the response variable.

Taguchi's parametric design of experiment (DoE) method also provides the identification and optimization of parameters, significance, and sensitivity [24]. Hence, this DoE has been widely used for the optimization of the process of varied fields including drug delivery [25-29], food packaging [30,31], treatment of wastewater from textiles [26-28], pulp and paper [32-37], biotechnology [38-41], electroplating [42,43], sputtering [44,45], nanotechnology [46-47].

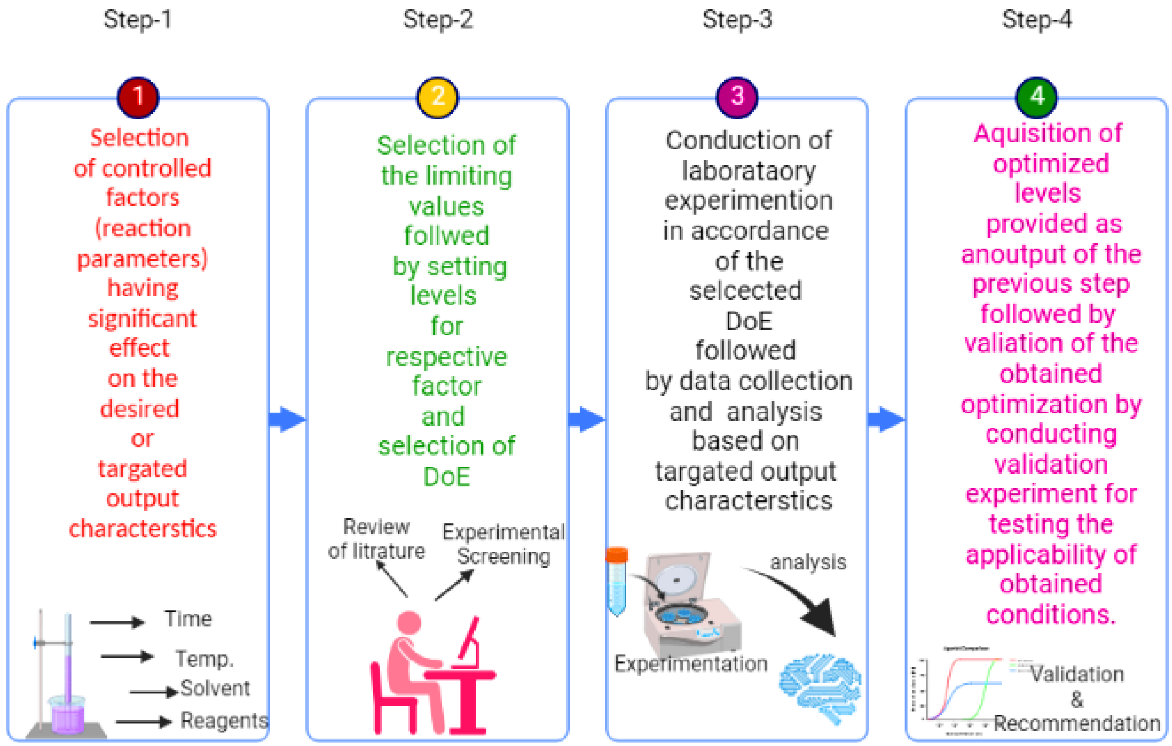


Figure 1. Schematic representation for the conduction of experimental study using DoE

Therefore, keeping in view, the importance of the fractional factorial design specially for resource-constrained projects, here in the present study a detailed step-by-step approach is discussed for DoE.

In our previous study, Taguchi's L16 orthogonal array was used for optimizing the reaction condition for synthesizing the quaternary ammonium derivative of guar gum with a high degree of substitution (DS) [48]. The present discussion focuses on acquiring the reaction conditions for the quaternization of DS.

Steps for conducting statistical analysis

DoE is a systematic approach for process optimization involving several steps as displayed in Figure 1 and mentioned in subsequent sections;

Identification of the targeted output or statement of the problem & selection of appropriate controlled factors and levels

Generally, the researchers conduct the experiments for two purposes; (i) to study the impact of the control factors or parameters on the final output, and (ii) to optimize the experimental conditions for targeted properties/characteristics in the final product.

Therefore, the experimenter must logically select the factors/parameters and their limits of variation along with the specific levels at which experimental runs will be carried out.

Detailed theoretical and practical knowledge of the process/reaction is required for this step. In selecting the controlling factor and their levels review of the literature or screening experiments or a combination of both may be taken into consideration as displayed in Figure 2.

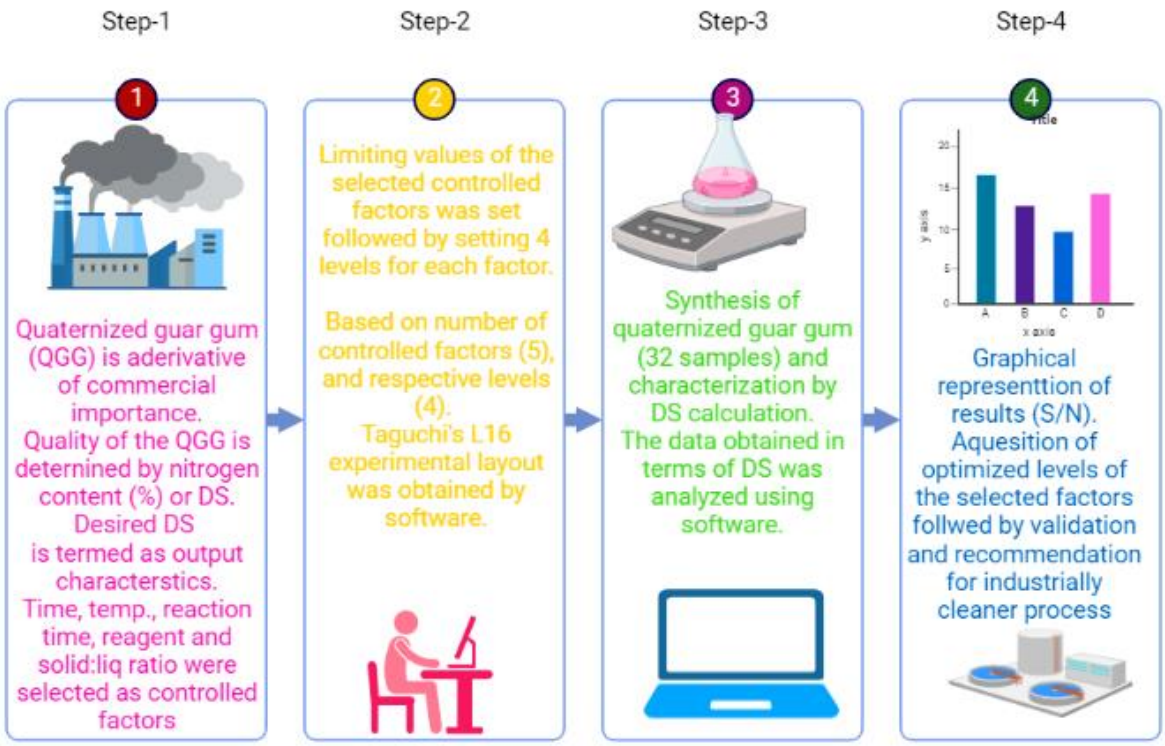


Figure 2. Schematic representation of the application of Taguchi's DoE for selected model study (quaternized guar gum of desired DS)

Selection of the suitable design of experiment (DoE)

If the first step is completed this step is relatively easy. Selection of DoE involves consideration of sample size (number of replicates) and selection of a suitable run order for the experimental trials. Generally, statistical software is applied for generating the DoE layout.

Based on the number of parameters and their levels, Taguchi's L16 (16 experiments) was selected for the quaternization of guar gum, and each experiment was carried out in a single replicate resulting in a total of 32 experiments. L16 layout is displayed in Table 2.

Table 1. Selected controlled factors and respective levels

S.no	Code of variable	Description of variable	Levels and assigned value			
			1	2	3	4
1	F1	Amount of NaOH (mol/AGU)	1.62	2.43	3.24	4.05
2	F2	Amount of CHPTAC (mol/AGU)	0.52	1.00	1.52	2.04
3	F3	Temperature (°C)	30	40	50	60
4	F4	Time (h)	1	2	3	4
5	F5	Gum: liquor ratio (w/v)	1:05	1:10	1:15	1:20

Performing the experiment

After generating the suitable DoE (L16) layout the next step is to perform the experiments by utilizing a suitable methodology by selecting the reaction conditions as suggested experimental layout. A total of 32 experiments were conducted to synthesize the quaternized guar gum followed by nitrogen estimation and DS calculation and the results are displayed in Table 2 (The experimental part of the study is already published by R. Tyagi *et al*., 2019 [48].

Data analysis

Next to the experimentation, step is the analysis of the data generated by determining the quantitative measurement of the targeted characteristics in the synthesized samples. As in the case of quaternization of guar gum, the targeted property was higher DS, and data was analyzed accordingly in the earlier experimental study. Now in the present segment, the data will be analyzed for obtaining the quaternized guar gum with lower is better. For the experiments conducted to optimize the process parameter to minimize any characteristic in the final product the smaller is better approach is utilized for which the S/N ratio formulate is expressed as;

$$\frac{S}{N} = -10.\log\left[\frac{1}{n}\sum_{i=1}^{n} Y_I^2\right] \quad (1)$$

where Yi is the achieved value in the experimental test and n is the number of tests. Equation (1) was used to calculate the S/N ratios of five factors and their levels for the fiber diameters.

$\frac{S}{N}$ Ratio graphs

The Taguchi-based methodology provides the results of the optimization study in the form of $\frac{S}{N}$. The graphs generated by the STATISTICA software are displayed in Figure 3. There are 5 graphs, one for each selected factor. The highest point or value of the generated graphs signifies the optimized level of the respectively controlled factor. For factor F1 (NaOH) the optimized level was 2nd for the coded value is 2.34 (mol/AHG). For factors F2 (CHPTAC) and F3 (Temp.) optimized level was 3rd for which coated values are 1.52 (mol/AGU), and 50 (°C). While for factors D (Time) and E (Gum: liquor) the optimized levels were 4th and 2nd respectively for which code values are 4 (Hour) and 1:20 (w/v) respectively. The optimized level of the selected factors along with their expected S/N ratio is displayed in Table 3.

Table 2. Experimental layout using Taguchi L_{16} (4^5) orthogonal array and degree of substitution (DS)

Replicate	F1	F2	Factor F3 Level	F4	F5	Designation (Sample Code)	Nitrogen (%)	DS
1	4	3	2	4	1	$1F1_4F2_3F3_2F4_4F5_1$	0.49	0.059
1	2	4	3	2	1	$1F1_2F2_4F3_3F4_2F5_1$	1.35	0.183
1	1	3	3	3	3	$1F1_1F2_3F3_3F4_3F5_3$	0.15	0.017
1	2	2	1	4	3	$1F1_2F2_2F3_1F4_4F5_3$	0.16	0.018
1	1	4	4	4	4	$1F1_1F2_4F3_4F4_4F5_4$	0.29	0.034
1	4	1	4	2	3	$1F1_4F2_1F3_4F4_2F5_3$	1.67	0.236
1	2	1	2	3	4	$1F1_2F2_1F3_2F4_3F5_4$	0.95	0.122
1	3	3	1	2	4	$1F1_3F2_3F3_1F4_2F5_4$	3.14	0.550
1	1	1	1	1	1	$1F1_1F2_1F3_1F4_1F5_1$	2.09	0.312
1	4	4	1	3	2	$1F1_4F2_4F3_1F4_3F5_2$	1.25	0.167
1	3	1	3	4	2	$1F1_3F2_1F3_3F4_4F5_2$	0.20	0.023
1	1	2	2	2	2	$1F1_1F2_2F3_2F4_2F5_2$	2.37	0.370
1	3	4	2	1	3	$1F1_3F2_4F3_2F4_1F5_3$	3.09	0.537
1	3	2	4	3	1	$1F1_3F2_2F3_4F4_3F5_1$	1.09	0.143
1	4	2	3	1	4	$1F1_4F2_2F3_3F4_1F5_4$	2.22	0.340
1	2	3	4	1	2	$1F1_2F2_3F3_4F4_1F5_2$	0.99	0.130
2	4	3	2	4	1	$2F1_4F2_3F3_2F4_4F5_1$	0.19	0.022
2	2	4	3	2	1	$2F1_2F2_4F3_3F4_2F5_1$	1.54	0.214
2	1	3	3	3	3	$2F1_1F2_3F3_3F4_3F5_3$	0.96	0.124
2	2	2	1	4	3	$2F1_2F2_2F3_1F4_4F5_3$	0.26	0.031
2	1	4	4	4	4	$2F1_1F2_4F2_4F4_4F5_4$	0.24	0.028
2	4	1	4	2	3	$2F1_4F2_1F3_4F4_2F5_3$	2.10	0.314
2	2	1	2	3	4	$2F1_2F2_1F3_2F4_3F5_4$	0.92	0.118
2	3	3	1	2	4	$2F1_3F2_3F3_1F4_2F5_4$	2.98	0.510
2	1	1	1	1	1	$2F1_1F2_1F3_1F4_1F5_1$	2.02	0.300
2	4	4	1	3	2	$2F1_4F2_4F3_1F4_3F5_2$	1.44	0.197
2	3	1	3	4	2	$2F1_3F2_1F3_3F4_4F5_2$	0.20	0.023
2	1	2	2	2	2	$2F1_1F2_2F3_2F4_2F5_2$	1.24	0.165
2	3	4	2	1	3	$2F1_3F2_4F3_2F4_1F5_3$	2.98	0.509
2	3	2	4	3	1	$2F1_3F2_2F3_4F4_3F5_1$	1.25	0.167
2	4	2	3	1	4	$2F1_4F2_2F3_3F4_1F5_4$	2.00	0.300
2	2	3	4	1	2	$2F1_2F2_3F3_4F4_1F5_2$	1.07	0.140

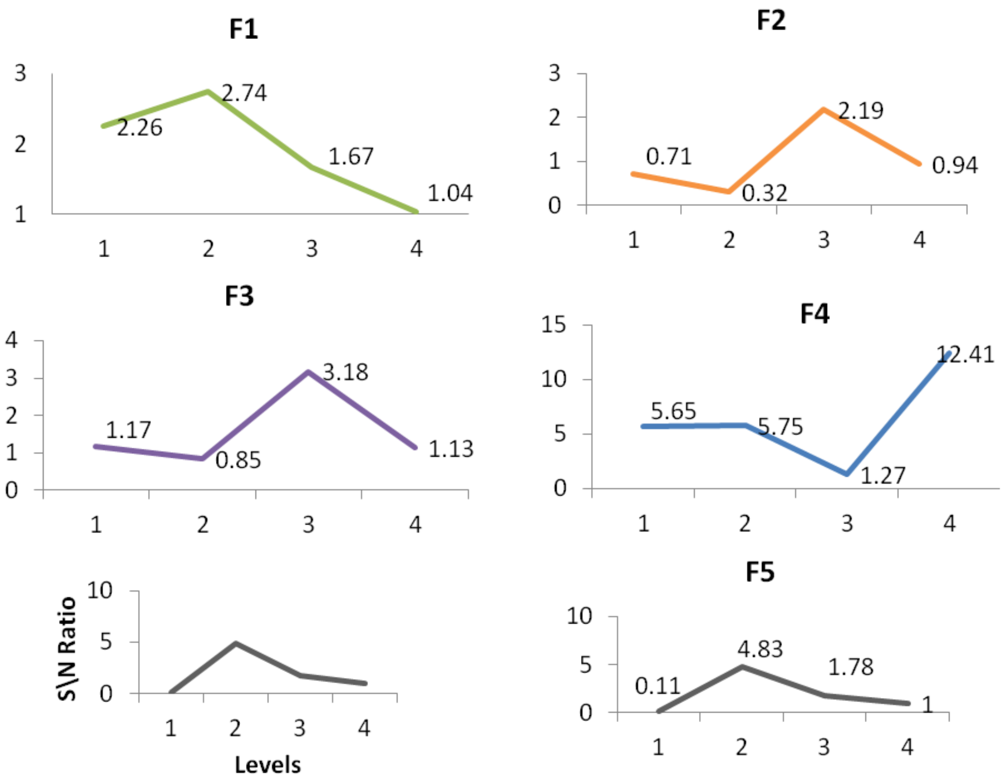


Figure 3. $\frac{S}{N}$ graph for the selected controlled factors F1, F2, F3, F4 and F5

Table 3. Energy parameters of the molecule VDB

Factor	Level	Effect of Size
F1	2	2.17029
F2	3	1.62029
F3	3	2.61484
F4	4	11.84720
F5	2	1.21257

Anova provides a quantitative result for the effect of the selected controlled factor on the targeted characteristics. In the present study, the p values greater than 0.05 will have a significant effect on targeted output. As the results displayed in Table 4, in the present study A and D were identified as significant factors for the tested characteristics.

Once the data have been analysed, the experiment must draw practical conclusions about the results and recommend a course of action. Graphical methods are often useful in this stage, particularly in presenting the results. Follow-up runs and confirmation testing should also be performed to validate the conclusions from the experiment.

Recommendations

Table 4. ANOVA

Factors	SS	df	MS	F	P
F1	121.904	3	40.6345	3.33608	0.046008
F2	40.145	3	13.3818	1.09864	0.378449
F3	97.997	3	32.6655	2.68183	0.081761
F4	1757.129	3	585.7095	48.08656	0.000000
F5	44.322	3	14.7739	1.21293	0.337093
Residual	194.885	16	12.1803		

Conclusions and Outlook

Optimization is one of the most frequently opted-in chemical processes. The present study provided a detailed explanation for the selection of DoE for optimizing the resource-constrained process. A detailed discussion is provided regarding various steps including selection and/or screening of controlled factors, selection of DoE, and experimentation followed by an explanation of the results. The paper will be helpful to researchers of a wide domain.

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Disclosure statement

The authors declare that they have no conflict of interest

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