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Active and collaborative learning approach in Unit Operations laboratory by Open-ended experiments using the concept of Design of Experiments (DOE)

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***Abstract*—Unit Operations is the hallmark course of Chemical Engineering and allied disciplines. This involves the separation of products by physical means such as Distillation, Extraction, Gas absorption, Crystallization, Evaporation, Filtration, etc. Unit Operations course is usually offered in the second year of the undergraduate program in Biotechnology. The conventional pedagogy practiced to date was not so effective in the attainment of certain Program Outcomes (PO). To address this issue, categorization of the laboratory was done into four types: Demonstration, Exercise, Structural inquiry, and Open-ended experiments was made. The experiments were carefully designed to address some of the major Technical and Professional outcomes. Assessment criteria for each kind of experiment were mapped to Program outcomes, Outcome elements, and Performance Indicators. This helps in the evaluation of the attainment of Program Outcomes more accurately.**

The present study deals with an active and collaborative learning approach in the Unit Operations laboratory by framing Open-ended experiments using the concept of Design of Experiments (DoE). Full Factorial Design(FFD) and Response Surface Methodology(RSM) concepts were used to study the effect of various parameters on Liquid-Liquid Extraction operation. The study helped in addressing the Program Outcome-4 defined by the National Board of Accreditation, which is related to conducting investigations of complex problems. The evaluation of the students' performance was done using rubrics-based methodology, and attainment of the graduate attributes addressed were documented. Quality students's feedback was taken to identify scope for further improvement in the teaching-learning process.

***Keywords*—Collaborative learning; Design of experiments; Open-ended experiment; Program outcome**

***JEET Category*— Research**

I. INTRODUCTION

Compared to a decade before, today's engineering Education is becoming more practical-oriented and hands-on activities are of prime importance in learning the fundamentals. The effectiveness of engineering education is influenced by the teaching and learning process adapted. The contribution of laboratory experiments in understanding the fundamental concepts and developing the research skills among students is enormous. A lot of studies are going on to effectively educate the undergraduate students; among those, one of the methodologies is to categorize laboratory experiments into four categories, namely: demonstration, exercises, structured inquiry, and open-ended experiments. These categories were well defined in developing the student's skills. The program outcome-4 can be well-matched with these categories. Demonstration and exercises help acquire the skills like conduction, obtaining required data, and analyzing data. Structured inquiry problems are developing the research skills in students by investigating a given problem. Open-ended experiments are ideal for giving exposure to designing experiments for a given problem situation. These open-ended experiments are designed to improve the problem-solving skills among students and to bring in a research culture. Program Outcome-4(Graduate Attribute), defined by the National Board of Accreditation, states that engineering graduates must have "an ability to design and conduct experiments, as well as to analyze and interpret data". The ability to conduct experiments, analyze and interpret data has been addressed by traditional laboratory courses, whereas the ability to design an experiment presents a new challenge (Jangali Satish *et al*.,2017).

In traditional laboratory courses, the experiments were of the exercise level wherein students conduct experiments in conventional mode (Eutimio Gustavo Fernandez. *et al*.,2013; Sharanappa, A. *et al*.,2018; Romero R.*et al*.,1995; Dahms, A.S *et al*.,2001; Shivalingsarj Desai. *et al*.,2016).

In the present work, an attempt has been made to bridge the gap between theory and practice and make students industry-ready

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through collaborative and experiential learning in the Unit Operations laboratory for undergraduate students of the Biotechnology program. In the present study, an open-ended experiment was performed using the Design of Experiments (DoE) approach to strengthen the ability of students to design and conduct experiments, analyze and interpret data. Through this open-ended experiment, students were involved in a team and investigated the effects of various process parameters on Liquid-Liquid Extraction Operation. Optimization of Process Parameters for Extraction of solute present in feed material using Response Surface Methodology (RSM) was done. Further, students analyzed the interaction effect among the various parameters affecting the extraction process (Talgar C.P, *et al.*, 2015; Sharanappa, A. *et al*.,2020).

MINITAB software was used to get the design matrix, regression model, Analysis of Variance (ANOVA), effects plots, response optimizer, surface and contour plots. The rubric-based assessment was done for the attainment of program outcome-4.

II. METHODOLOGY

A. Design of Unit Operations Laboratory

The Unit Operations Laboratory was designed taking into consideration of inputs from industry stakeholders. The Laboratory experiments were categorized into four categories (Demonstration, Exercise, Structured inquiry, and Open-Ended Experiment). Accordingly, the present study was a part of Open-ended experiments wherein the DoE approach was implemented to enhance student's ability to design, conduct, analyze and interpret data (Sharnappa A.*et al*.,2020; Zaiton Haron. *et al*.,2013; Anil Shet.*et al*.,2017; Satish Jangali and Vinayak Gaitonde.,2020). The program outcomes addressed through this activity are PO-4 (Conduct Investigation of complex problems, PO-10 (Communication: Communicate effectively through writte and mode), Program Specific Outcome: PSO-13 (Proficiency of good laboratory practices-GLP and Standard Operating Procedures-SOP).

B. Implementation of the Laboratory

The present study was carried out for second-year undergraduate students of engineering in Biotechnology for Unit Operations Laboratory.

About the Laboratory**:**

Unit Operations is fundamental in Chemical Engineering and allied disciplines which involve separation of products by physical means such as Distillation, Extraction, Gas absorption, Crystallization, Evaporation, Filtration, etc.

The laboratory comprises experiments on several unit operations involving heat transfer, mass transfer, and momentum transfer operations. Unit operations involve a physical change or chemical transformation such as separation, extraction, crystallization, evaporation, distillation, filtration, polymerization, isomerization, and other reactions. A process may require many unit operations to obtain the desired product from the starting materials or feedstocks.

Extraction is one of the important unit operations widely used in the separation of vital products from the reaction mixture. An Open-ended experiment was designed to study the factors affecting Liquid-Liquid Extraction operation.

Modus Operandi of implementation:

The execution of the Unit Operations Laboratory in three modules is represented in Fig 1.

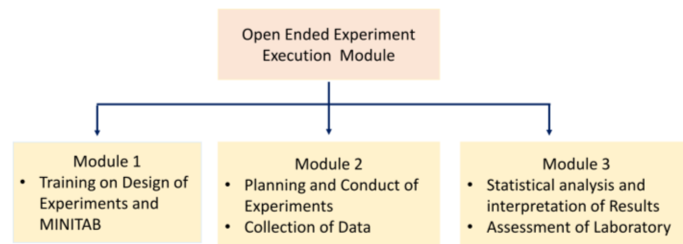

Fig 1. The open-Ended Experiment Execution module

Module-1: Training: Students were trained on Standard Operating Procedures (SOP) of different types of equipment involved in Unit Operations. Briefing on Good Laboratory Practices(GLP) pertaining to Unit Operations Laboratory. Students were trained in various aspects of DoE such as Factorial Experiments, the Design matrix, RSM, Main effects and Interaction effects, Regression model, ANOVA, Optimizer. Students were trained on the usage of statistical software MINITAB.

Module-2: Conduction of Experiment & Collection of Data. The total strength of the class was 55, and groups of 5 students were formed to conduct the open-ended experiment. Each group has freedom of selecting different feed and solvents systems, factors affecting extraction process and their levels. The results obtained by one group are presented here for illustration.

Module-3: Analysis & Interpretation of Results.

III. ASSESSMENT

A. Assessment of Open-Ended Experiment

The rubrics-based assessment was carried out as per the rubric parameter (Laxmikant Patil. *et al*., 2021; Akhil Sachan. *et al*.,2019; Anil Shet.*et al*.,2017; Zabin Bagewadi. *et al*.,2020) shown in Table I and Table II.

The collaborative and individual efforts of students were assessed through viva-voce examination.

TABLE I

MAPPING OF RUBRIC PARAMETERS WITH PERFORMANCE INDICATORS FOR PO-4

Graduate Attribute: Conduct investigation of complex problems **PO4:** An ability to design & conduct experiments, as well as analyze data.	
Rubrics Parameters	**PI Addressed**
Identification of Problem/ parameter	4.1.1: Define a problem to investigate with its scope and importance.
Selection of appropriate procedure	4.1.2: Identify and apply the relevant experimental procedure for a defined problem
Conduct of experiment	4.1.3: Use appropriate analytical instruments to carry out the experiments
Data Collection & representation	4.3.1: Use appropriate procedures, tools, and techniques to collect and analyze data
Analysis of data	4.3.2: Critically analyze data for trends and correlations, stating possible errors and limitations.
Interpretation of data	4.1.4: Correlate the experimental outcomes with underlying theoretical concepts and principles
Conclusion	4.3.4: Synthesize information and knowledge about the problem from the raw data to reach appropriate conclusions

TABLE II

MAPPING OF RUBRIC PARAMETERS WITH PERFORMANCE INDICATORS FOR PO-10 AND PSO-13

Graduate Attribute: Communication **PO-10:** An ability to communicate effectively.	
Rubrics Parameters	**PI Addressed**
Journal Write up	10.1.2: Produce clear, well-constructed, and well-supported written engineering documents.
PSO-13 Program Specific Outcome	
Following SOP & GLP	13.2.2: Follow standard operating procedures adhering to laboratory guidelines.

B. *Implementation of Open-ended experiment*

Students in a team were given an open-ended problem to analyze the effects of process parameters on the Extraction operation. The students need to perform the above activity using the following steps.

- Selection of the significant process parameters/factors and their levels
- Planning of experiments using design matrix.
- Conduct experiments as per design matrix & record of the response.
- Developing regression model
- Analyzing the experimental data
- Checking the adequacy of the developed models
- Validation of the developed models.
- Interpreting the experimental results

Designing the experiment

Full Factorial Design (FFD), consisting of 3 factors, each at three levels, was considered. Replication reduces variability in experimental results and increases the confidence level of the researcher. The experiment was carried out with three replications. The process parameters and their levels identified are mentioned in Table III. Open-Ended Experiment on Extraction was performed by maintaining other process parameters constant (Kwang-Min, L. *et al*.,2006; Deepak Yaraguppi. *et al.*,2016). The process parameters maintained constant were temperature (ambient room temperature), feed sample (Benzene-Acetic acid solution), and solvent (distilled water).

TABLE III

FACTOR AND LEVELS

Process Parameters/ Factors	**LEVELS**		
	Low (-1)	**Medium (0)**	**High (1)**
Quantity of Solvent (ml)	100	200	300
Time of extraction (min)	10	20	30
Agitation Speed (RPM)	50	100	150

The experiments were planned as per the FFD matrix (Table IV) generated by MINITAB software.

TABLE IV

EXPERIMENTAL DESIGN MATRIX

Run Order	Qty of Solvent	Time of Extraction	Agitation speed (RPM)
1	-1	-1	-1
2	-1	0	-1
3	-1	+1	-1
4	0	-1	-1
5	0	0	-1
6	0	+1	-1
7	+1	-1	-1
8	+1	0	-1
9	+1	+1	-1
10	-1	-1	0
11	-1	0	0
12	-1	+1	0
13	0	-1	0
14	0	0	0
15	0	+1	0
16	+1	-1	0
17	+1	0	0
18	+1	+1	0
19	-1	-1	+1
20	-1	0	+1
21	-1	+1	+1

22	0	-1	+1
23	0	0	+1
24	0	+1	+1
25	+1	-1	+1
26	+1	0	+1
27	+1	+1	+1

Conduct of experiment:

Based on the experimental plan, the students conducted experiments with three replications for each trial run. The percentage recovery of solute by extraction (response) was measured by Titrimetric analysis and has been recorded for each experimental run. The regression coefficients were determined, and the adequacy of the model was checked by performing an analysis of variance (ANOVA). Table V shows the experimental values of the response.

TABLE V
EXPERIMENTAL RESPONSES

Run order	Percent Recovery Avg ± SD
1	41.66 ± 1.21
2	64.69 ± 1.64
3	48.94 ± 1.70
4	75.23 ± 2.06
5	85.17 ± 1.21
6	72.44 ± 0.67
7	59.48 ± 1.62
8	71.43 ± 0.59
9	49.82 ± 1.33
10	53.88 ± 1.58
11	64.48 ± 0.63
12	47.25 ± 0.94
13	80.38 ± 1.25
14	89.84 ± 3.16
15	77.10 ± 0.29
16	56.33 ± 1.04
17	65.97 ± 1.47
18	53.27 ± 1.48
19	41.92 ± 1.03
20	59.73 ± 2.42
21	42.63 ± 0.31
22	71.81 ± 1.17
23	84.26 ± 0.67
24	66.59 ± 1.04
25	54.67 ± 1.79
26	67.42 ± 1.24
27	40.41 ± 0.43

The result showed an average optimum percent recovery of 89.84 at experimental run order 14th, where all three process parameters were at their mid-levels. The lowest percent recovery observed was 40.41 at 27^{th} run order, where all process parameters were at a high level(Douglas C. Montgomery.*et al.*,2019).

Analysis and Interpretation of Results

The Optimization of response (Percent Recovery) was carried out using Response Surface Methodology. Students used MINITAB software to analyze the data regarding the significance of the regression model, main effect, interaction effect, effect plots, surface plots, Contour plots, Residual plots, and Response Optimizer.

The postulation of Regression Model

A regression model is an equation relating to the process parameters and the response. The assumed regression models considering three-factor interaction for the current investigation can be written as:

$$Y_i = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{11} A*A + \beta_{22} B*B + \beta_{33} C*C + \beta_{12} A*B + \beta_{13} A*C + \beta_{23} B*C$$

where, Y_i –Response (Percentage recovery of solute by Extraction process), A- Qty of Solvent (ml), B-Time of extraction(min), C- Agitation speed (RPM), $\beta_0, \beta_1, \beta_2, \ldots, \beta_{23}$ are the regression coefficients, A*B, A*C, and B*C are the interaction terms.

Regression Equation in Un-Coded Units:

The relationship between response and process parameters was predicted by RSM quadratic model in terms of un-coded units, as shown below.

Percent Recovery = -94.47 + 1.0261 Qty of Solvent
+ 6.543 Time of Extraction + 0.3754 RPM
- 0.002343 Qty of Solvent*Qty of Solvent
- 0.15122 Time of Extraction*Time of Extraction
- 0.001749 RPM*RPM
- 0.002362 Qty of Solvent*Time of Extraction
- 0.000121 Qty of Solvent*RPM
- 0.00227 Time of Extraction*RPM

TABLE VI
ANOVA TABLE

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	15563.6	1729.29	189.22	0.000
Linear	3	965.0	321.67	35.20	0.000
Qty of Solvent	1	479.1	479.15	52.43	0.000
Time of Extraction	1	226.8	226.84	24.82	0.000
RPM	1	259.0	259.02	28.34	0.000

Square	3	14338.4	4779.45	522.96	0.000
Qty of Solvent*Qty of Solvent	1	9878.2	9878.19	1080.86	0.000
Time of Extraction * Time of Extraction	1	4116.1	4116.11	450.38	0.000
RPM*RPM	1	344.1	344.06	37.65	0.000
2-Way Interaction	3	260.3	86.75	9.49	0.000
Qty of Solvent*Time of Extraction	1	200.9	200.91	21.98	0.000
Qty of Solvent*RPM	1	13.1	13.08	1.43	0.236
Time of Extraction*RPM	1	46.3	46.27	5.06	0.028
Error	71	648.9	9.14		
Lack-of-Fit	17	488.2	28.72	9.65	0.000
Pure Error	54	160.7	2.98		
Total	80	16212.5			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
3.02311	96.00%	95.49%	94.85%

From Table VI, the following inferences were drawn.

- The regression model is significant (P-value < 0.05 level of significance)
- All the main effects and interaction effects were found to be significant except the interaction Qty of Solvent*RPM (P-value < 0.05 level of significance).
- The adequacy of the model can be verified by the coefficient of determination. From the regression analysis, the coefficient of determination(R^2) was found to be 96% which is very close to 1. This indicates 96% of total variability is explained by the regression model. The adjusted R^2 value is 95.49%, and the predicted R^2 value is 94.85%.

From Fig 2, it is evident that all the main effects and interaction effects were found to be significant excluding the AC interaction (i.e. Qty of Solvent*RPM).

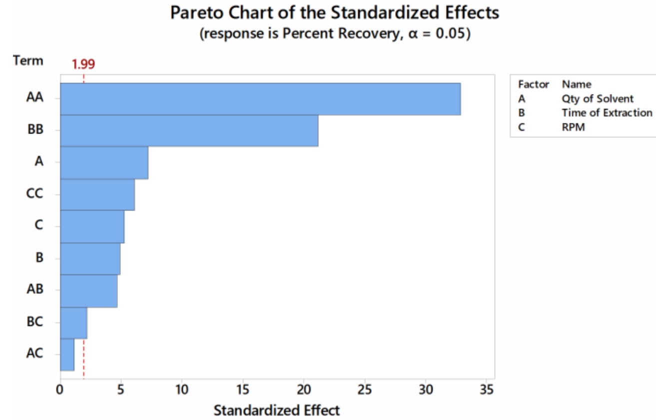

Fig 2. Pareto Chart

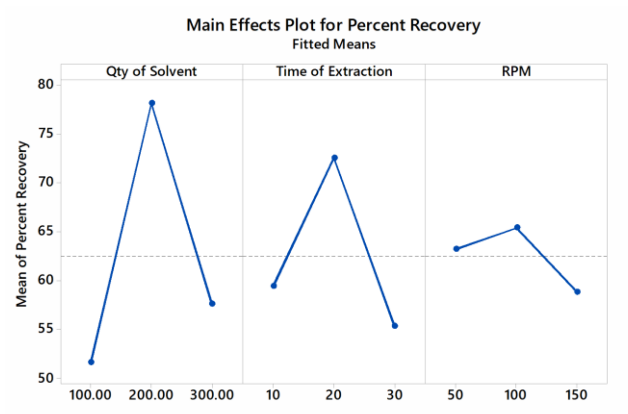

Fig 3. Main effect plot

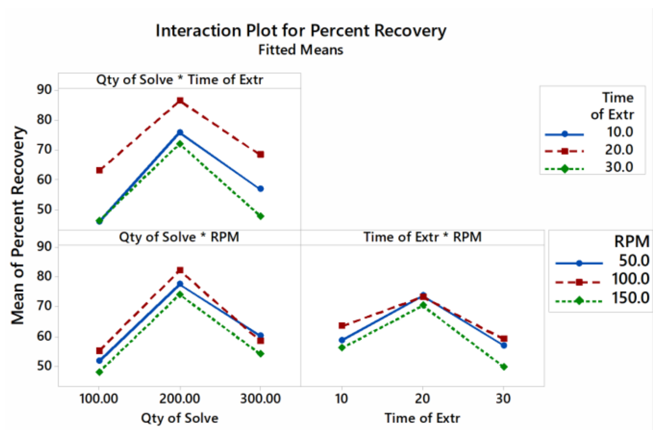

Fig 4. Interaction plot

From main effect plots shown in Fig 3, it can be witnessed that as the quantity of solvent increases from 100ml to 200ml, the percentage recovery increases from 52% to 78% and then decrease to 58% as the quantity of solvent is further increased to 300ml. Similarly, as the time of extraction increases from 10minutes to 20minutes, the percentage recovery increases from 58% to 72% and then decreases to 54% as the time of extraction is further increased to 30minutes. Similarly, as agitation speed (RPM) increases from 50rpm to 100rpm, the percentage recovery increases from 63% to 65% and then further decreases to 57% as the agitation speed is further increased to 150rpm, thus indicating the nonlinear relationship between parameters and the response (Nadiya, R. *et al.*,2016;

Eswaraiah.C, 2015). From the Interaction Plot (Fig 4), it can be inferred that there is little interaction among the process parameters.

3D surface plots (Fig 5.) were plotted to study the interaction effects of Process parameters on response. In 3D surface plots, the vertical axis represents response (Percent Recovery), and two horizontal axes represent the uncoded levels of two independent process parameters by keeping other variables at their control level.

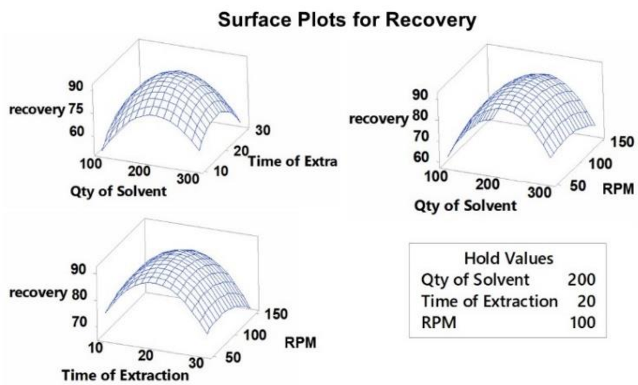

Fig 5. Surface Plot

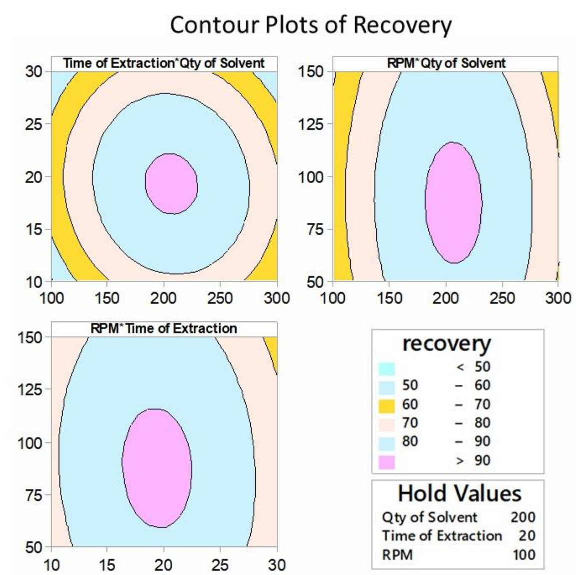

Fig 6. Contour Plot

From the contour plot (Fig 6), it can be observed that more than 90% of the recovery is at the mid-level of process parameters.

Experimental Model Validation

Experimental validation of the model equation was tested by carrying out experiments under the optimum settings. The optimum settings from RSM optimizer are presented in Fig 7.

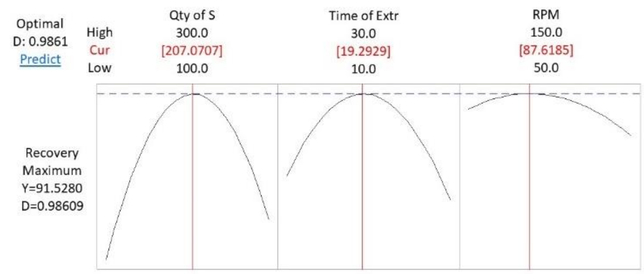

Fig.7 RSM Optimizer

TABLE VII
OPTIMAL PARAMETER SETTINGS

Factor	Setting	Model predicted value	Experimental value
Quantity of solvent(ml)	207	91.53	90.39
Time of Extraction(min)	19		
Agitation Speed (rpm)	88		

With the Parameter settings mentioned in Table VII, experiments were performed in triplicates. Percent recovery was found to be 90.39, which is in good agreement with the predicted value of 91.53 from the regression model. Hence the validity of the model was proved.

C. Student Feed Back

A feedback survey was conducted and a sample copy of the questionnaire is shown in Table VIII. For each question, 0-10 scale was used, 0 being the lowest score and 10 being the highest. 52 students participated in the study.

TABLE VIII
QUESTIONNAIRE FOR STUDENT FEEDBACK

Sl. No	Questions
1	Implementation of the Design of Experiment (DoE) tool enhanced the ability to design & conduct experiments, analyze & interpret the results, and synthesize information to provide valid conclusions.
2	Open-ended experiments were helpful in a deeper understanding of important concepts of Unit Operation through experiential learning and strengthened the domain knowledge.
3	Training on statistical software was helpful in the analysis of data and interpretation of results.
4	Adequate proficiency of GLP and SOP was obtained during the execution of this laboratory.
5	The activity helped in improving verbal and written communication skills.

IV. Results

Implementing the DoE approach in the Open-Ended Experiment in Unit Operations Laboratory through collaborative learning was instrumental in strengthening domain knowledge, usage of statistical software, analysis, interpretation & decision-making skills. The evaluation of attainment of POs by mapping the rubrics parameters with PI as shown in table I is carried out. The attainment of various PI's on a scale of 1 to 10 was measured and is presented in Fig 8.

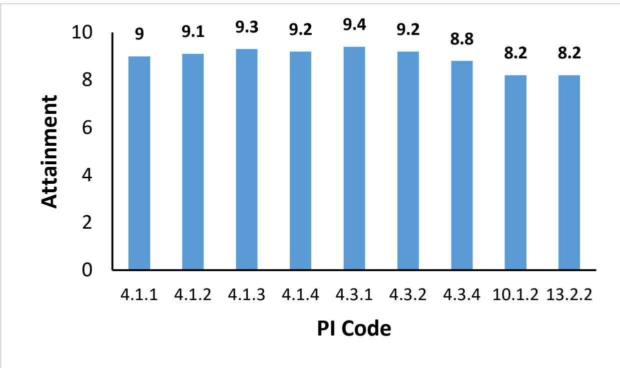

Fig 8. Attainment of program outcomes and performance indicators

The attainment for design & conduct of experiments, as well as analyze and interpret data (PO-4), was good, which indicates that the implementation of DoE enhanced skills such ability to design, conduct, analyze and interpret the results. However, there is scope for improvement in the area of Communication (PO-10), such as report writing and follow of SOP & GLP (PSO-13).

The feedback of students on the implementation of the DoE module in the Open-Ended Experiment was analyzed. The results are shown in Fig 9. The feedback of the students revealed that the framework of DoE in the Open-Ended experiment of Unit Operations Laboratory was very helpful in enhancing the domain skill-sets and gave them experiential learning. However, there is scope for improvement in the area of SOP & GLP and communication Skills which need to be further improved in the next cycle

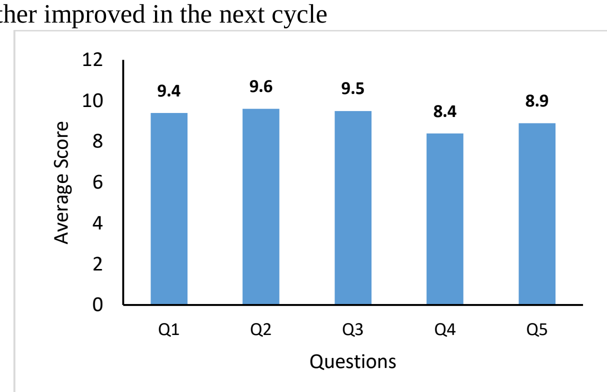

Fig 9. Students' feedback

The students expressed satisfaction in terms of having acquired the in-depth knowledge of DoE concepts such as identification of process parameter and their levels, planning and execution of experiments as per the design matrix, ANOVA, running the RSM optimizer, interpretation of the graphs, and the results to arrive at the valid conclusion in open-ended experiments.

V. Conclusions

From present study, it can be inferred that, the implementation of open-ended experiment in Unit Operations laboratory through active and collaborative learning proivde an independent, innovative and creative platform to execute the experiments. DoE approach facilitates students to plan & conduct the experiments effectively. Analyzing the data and subsequently interpreting the results, the students could acquire a deeper understanding of concepts of Unit Operations laboratory through experiential learning, indicating the better attainment of PO-4. Open ended experiment makes reliable assessments possible due to the use of rubrics in assessment of lab report, team work and practical skills. The students' feedback indicates that the use of DoE enhances the domain skill-sets and usage of statistical software. However, there is a scope for improvement in area like Communication (PO-10) and Following SOP & GLP (PO-13).

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