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Strengthening the Attainment of Major Competencies of Program Outcomes through Open-ended Experiment using Statistical Designs

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***Abstract*—Unit operations are very essential part of the chemical engineering and allied disciplines; and hence, basic knowledge about the principles and equipment used for various unit operations is mandatory for process engineer. Unit operations course introduces the foundation of process engineering concepts at the second year of undergraduate program of Bachelor of Engineering in Biotechnology. This course mainly focuses on the most essential unit operations utilized in Chemical and allied process industries such as mass transfer, heat transfer, momentum transfer and mechanical operations. The conventional teaching approach practiced so far in Unit Operations Laboratory course was not so effective in the attainment of certain specific core competencies of Program Outcomes (POs). To tackle this challenge, we introduced a system of classifying laboratory experiments into four distinct categories: Demonstration, Exercise, Structural Inquiry, and Open-ended experiments. These categorized experiments were thoughtfully crafted to target various significant technical and professional outcomes such as Investigations of Complex Engineering Problems, Communication, Life-long learning, Follow of Standard Operating Procedure (SOP) and Good Laboratory Practices (GLP). The evaluation criteria for each type of experiment were aligned with program outcomes, competencies, and performance indicators. This alignment proved highly beneficial in achieving a more precise assessment of program outcomes. This study focuses on implementing an interactive and collaborative learning approach in the Unit Operations laboratory course.**

This is accomplished through the integration of open-ended experiments that apply the principles of Statistical Design of Experiments. More precisely, the study employs Central Composite Design (CCD) and Response Surface Methodology (RSM) concepts to examine how various factors influence the Leaching (Solid-Liquid Extraction) process. The leaching process is most important unit operations used in the extraction of substances from solids. The study helped in addressing several important competencies pertaining to the Program Outcomes- PO-4: Demonstrating the capacity to design, execute experiments, and analyze data; PO-10: Exhibiting effective communication skills; PO-12: Embracing lifelong learning; PSO-13: Adhering to Standard Operating Procedures (SOP) and Good Laboratory Practices (GLP). The assessment of student performance employed a rubric-based approach, and the attainment of program outcomes was documented. It was observed that, open-ended experimental approach, led to the improvements in the attainment of PO's in terms of competencies and performance indicators. To enhance the teaching-learning process, formal and anonymous feedback was gathered from students to identify areas of improvement and deficiencies.

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***Keywords*—Central Composite Design; Collaborative learning; Design of experiments; Open-ended experiment; Program outcome; Response Surface Methodology;**

***JEET Category*—Research**

I. INTRODUCTION

Modern engineering education has undergone a shift towards increased practicality with significant emphasis on hands-on activities to master fundamental concepts. The effectiveness of engineering education is impacted by the methods adopted in the process of teaching learning. Laboratory experiments play a vital role in enhancing several specific core competencies and fostering research capabilities among students. Various studies are actively exploring ways to enhance the education experience of undergraduate students. One such approach involves classifying laboratory experiments into four distinct categories: demonstrations, exercises, structured inquiries and open-ended experiments. These classifications have been thoughtfully formulated to nurture students' competencies. Demonstration and exercise kind of experiments play a pivotal role in developing skills such as execution of experiment, data acquisition and data analysis. Structured inquiry type experiment contributes to the enhancement of students' research capabilities by prompting them to investigate the defined problems. Open-ended experiments are particularly valuable in providing students with an opportunity to design experiments tailored to specific problem scenarios. These experiments are deliberately designed to enhance students' problem-solving skill and to cultivate a research-oriented mindset. With the implementation of this categorization approach, students' aptitude in problem-solving and thinking is anticipated to flourish.

In conventional laboratory courses, experiments primarily consisted of exercises where students conducted experiments in a conventional manner (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; Laxmikant R Patil et al.,2023). This study introduces an open-ended experiment conducted using the Design of Experiments (DoE) approach, intended to enhance students' capabilities in designing, conducting, analyzing, and interpreting experiments. Through this open-ended experiment, students worked collaboratively in teams to explore the effects of various process parameters on the Solid-Liquid Extraction Operation. The optimization of process parameters for solute extraction from feed material was carried out using Response Surface Methodology (RSM). Furthermore, students examined the interaction effects among the different parameters influencing the extraction process (Talgar C.P, et al., 2015; Sharanappa, A. et al., 2020; Jangali Satish *et al*.,2017). The MINITAB software was employed to generate the design matrix, regression model, conduct Analysis of Variance (ANOVA), create effects plots, utilize response optimization tools, and generate surface and contour plots. The assessment of student attainment in relation to program outcomes was performed using a rubric-based approach.

2. Methodology

The laboratory experiments were classified into four distinct 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 (Sharanappa A.*et al*.,2020; Zaiton Haron. *et al*.,2013; Anil Shet.*et al.*,2017; Satish Jangali and Vinayak Gaitonde.,2020).

This activity addressed the program outcomes PO-4 (Investigate complex problems), PO-10 (Communication), PO-12 (Lifelong learning), Program Specific Outcome: PSO-13 (Adherence to good laboratory practices-GLP and Standard Operating Procedures-SOP). The present study was carried out for students of undergraduate program in Biotechnology Engineering.

About the Unit Operations Laboratory

Unit Operations constitutes an essential fundamental course within the domain of Chemical Engineering as well as its allied disciplines. They are crucial for processes that involve the physical separation of products, using methods like Crystallization, Extraction, Distillation, Gas Absorption, Evaporation and Filtration. Often, multiple unit operations are necessary in a sequence to transform the starting materials/feedstock into the desired end product. This Unit Operations laboratory includes experiments focusing on a range of operations that involve heat transfer, mass transfer, momentum transfer and mechanical processes. Extraction stands out as a significant unit operation that finds extensive use in separating valuable products from reaction mixtures. To delve into the intricacies of Solid-Liquid Extraction (Leaching), an open-ended experiment was devised to investigate the factors influencing this operation.

Mode of Execution

The open-ended experiment was executed in four phases.

Phase 1: Training
- Training on SOP and GLP
- Hands on training on implementation of DoE using statistical software

Phase 2: Execution of Open-ended experiment
- Planning of Experiments (Identification of process parameters and their levels, Design matrix)
- Conduct of experiments as per the plan.
- Data collection

Phase 3: Analysis and documentation
- Detailed analysis of data (Regression model, ANOVA, optimizer, surface and contour plots, etc.,)
- Report writing

Phase 4: Rubric based Evaluation
- Presentation of the findings of the open-ended experiment through Power point presentation and viva voce.

Training: Before the commencement of Open-ended experiment, students were trained on SOP and GLP to be followed in Unit Operations laboratory. Further students were trained in various fundamental concepts of Design of Experiments such as Full and Fractional Factorial Experiments, Design matrix, RSM, Main effects and Interaction effects, Regression model, ANOVA, Contour and Surface plots, Optimizer (Douglas C. Montgomery.*et al*.,2019). Students were provided with hands on training on the usage of statistical software.

Execution of Open-ended experiment: Conduction of Experiment & Collection of Data. The class comprised a total of 45 students, and teams consisting of 5 students each were established to execute an exploratory experiment. Each group has liberty to opt for different feed and solvents systems, various factors affecting extraction process and their corresponding levels. The result obtained by one of the groups is presented here for illustration.

Analysis & Documentation: Detailed statistical analysis of experimental data such as ANOVA, t-Test, F-Test, Regression analysis, analysis of main effect plot, interaction effect plot, surface and contour plots is performed. Report writing of open-ended experiment in the specified format is done.

Evaluation: Presentation of the findings of the open-ended experiment through Power point presentation and viva voce. The collaborative and individual efforts of students were assessed through viva-voce examination.

3. Assessment

A. Assessment of Open-Ended Experiment

The rubrics-based assessment was carried out for each PO, considering the competency and performance Indicator (PI) addressed (Vinayak Kulkarni. *et al*.,2017; Laxmikant Patil.*et al*., 2021; Akhil Sachan. *et al*.,2019; Anil Shet.*et al*.,2017; Praveen M et al.,2017; Zabin Bagewadi. *et al*.,2020; Gururaj N Bhadri et al.,2022). Mapping of Competency with Performance Indicators is shown in Table I.(For example 4.1.2 refers to Program Outcome '4', Competency '1' and Performance Indicator '2').The collaborative and individual efforts of students were assessed through presentation and viva-voce examination.

Table I: Mapping of Competency with Performance Indicators

Graduate Attribute: *Conduct Investigation of complex problems:* Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data and synthesis of the information to provide valid conclusions. **PO-4:** Ability to design & conduct experiments, as well as analyze data
Competency-4.1: Demonstrate ability to conduct investigations of technical issues consistent with their level of knowledge and understanding
PI Addressed
4.1.1: Define a problem to carry out investigation with its scope and importance
4.1.2: Identify and apply the relevant experimental procedure for a defined problem
4.1.3: Use appropriate analytical instruments/software tools to carry out the experiments
4.1.4: Correlate the experimental outcomes with underlying theoretical concepts and principles.

Competency-4.2: Demonstrate ability to design experiment to solve open ended problem
PI Addressed
4.2.1: Design and develop experimental flow charts and specify appropriate equipment for the given open-ended problem.

Competency-4.3: Demonstrate ability to critically analyze data to reach a valid conclusion
PI Addressed
4.3.2: Critically analyze data for trends and correlations, stating possible errors and limitations
4.3.3: Represent data in tabular and graphical form for data analysis
4.3.4: Synthesize information and knowledge about the problem from the raw data to reach appropriate conclusion

Graduate Attribute: *Communication:* Communicate effectively on complex engineering activities with the engineering community and with the society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions **PO-10:** Ability to communicate effectively
Competency-10.1: Demonstrate an ability to comprehend technical literature and document project work
PI Addressed
10.1.2: Produce clear, well-constructed, and well-supported written engineering documents.

Graduate Attribute: Life-long learning: **PO-12:** Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change
Competency-12.1: Demonstrate conceptual understanding of concepts of Design of Experiments and its applications
PI Addressed
12.1.1: Use appropriate tools, techniques of DOE based on the defined problem.
PSO-13: Following SOP and GLP
Competency-13.2: Demonstrate proficiency of good laboratory practices in terms of accuracy and precision, safety, ethics and reproducibility and able to follow standard operating procedures
PI Addressed
13.2.2: Following Standard Operating Procedure adhering to laboratory guidelines.

B. Implementation of Open-ended experiment

Designing the experiment

Central Composite Design (CCD), consisting of 3 factors, each at five levels, was considered. All these experiments were carried in duplicates. The process parameters and their levels identified are mentioned in Table II. Open-Ended Experiment on Solid-Liquid Extraction (Leaching) was performed by maintaining other process parameters constant (Kwang-Min, L. *et al*., 2006; Deepak Yaraguppi.*et al*.,2016; Douglas C. Montgomery.*et al*., 2019, Claire F. Komives, 2014).

Table II**.** Factor and levels

Process Parameters/ Factors	**LEVELS**				
	Lowest (-1.68)	**Low (-1)**	**Medium (0)**	**High (+1)**	**Highest (+1.68)**
Solvent to feed ratio (SFR)	1.318	2	3	4	4.682
Temperature(^{0}C)	33.182	40	50	60	66.818
Agitation speed (AS) (rpm)	115.910	150	200	250	284.09

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

Conduct of experiment:

Experiments were conducted by the students as per the experimental plan with two replications. The percentage recovery of solute by Solid-Liquid extraction (Leaching) was measured by acid- base titration method and has been recorded for each experimental run. The regression coefficients were determined, and the model adequacy was checked by performing an analysis of variance. Table III shows the CCD design matrix along experimental values of the response.

Table III. Experimental Design Matrix with response

Run Order	SFR	Temp	AS	Percent Recovery	
				Rep1	Rep2
1	-1	-1	-1	39.0	39.8
2	1	-1	-1	64.0	65.3
3	-1	1	-1	28.8	29.4
4	1	1	-1	67.7	69.0
5	-1	-1	1	40.6	41.5
6	1	-1	1	49.9	50.9
7	-1	1	1	74.6	76.1
8	1	1	1	70.6	72.0
9	-1.68	0	0	29.6	30.2
10	1.68	0	0	55.2	56.3
11	0	-1.68	0	59.8	61.0
12	0	1.68	0	67.7	69.0
13	0	0	-1.68	72.5	73.9
14	0	0	1.68	71.4	72.8
15	0	0	0	71.4	72.8
16	0	0	0	78.2	79.8
17	0	0	0	83.8	85.5
18	0	0	0	75.5	77.0
19	0	0	0	85.1	86.8
20	0	0	0	83.2	84.9

Data 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 Regression Model

The process parameters and the response are well connected by a quadratic regression model considering two factor interaction and is written as

$$y_i = b_0 + b_1 A + b_2 B + b_3 C + b_{11} A * A + b_{22} B * B + b_{23} C * C + b_{12} A * B + b_{13} A * C + b_{23} B * C$$

where, y_i –Response (Percentage recovery of solute by leaching process), A-Solvent to Feed ratio, B-Temperature (^{0}C), C-Agitation speed (RPM), b_0, b_1, . . . , b_{23} are the regression coefficients, A*B, A*C, and B*C are the interaction terms.
The predicted RSM quadratic model in terms of uncoded units is as below:

PERCENT RECOVERY= -254.1 + 121.8 SFR + 3.78 TEMP + 0.286 AS - 14.061 SFR*SFR - 0.06433 TEMP*TEMP - 0.001408 AS*AS + 0.008 SFR*TEMP - 0.1479 SFR*AS + 0.01543 TEMP*AS

Table IV. ANOVA table

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	10960.6	1217.85	42.69	0.000
Linear	3	2614.6	871.52	30.55	0.000
SFR	1	1879.8	1879.77	65.90	0.000
TEMP	1	559.3	559.26	19.61	0.000
Agitation Speed	1	175.5	175.51	6.15	0.019
Square	3	6518.7	2172.91	76.18	0.000
SFR*SFR	1	5698.4	5698.42	199.77	0.000
TEMP*TEMP	1	1192.9	1192.93	41.82	0.000
AS*AS	1	357.0	357.03	12.52	0.001
2-Way Interaction	3	1827.3	609.11	21.35	0.000
SFR*TEMP	1	0.1	0.10	0.00	0.952
SFR*AS	1	874.6	874.55	30.66	0.000
TEMP*AS	1	952.7	952.69	33.40	0.000
Error	30	855.8	28.53		
Lack-of-Fit	5	537.0	107.40	8.42	0.000
Pure Error	25	318.8	12.75		
Total	39	11816.4			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
5.34090	92.76%	90.59%	86.78%

The ANOVA results (Table IV) indicated a highly significant overall model (p-value <0.0001), as reflected in the substantial F-value. The R^2 and adjusted determination coefficient (Adj R-sq) exceeded 0.9, suggesting a strong correlation between the experimental and predicted values.

Fig 1 shows the standardized effects for the factors through Pareto chart. From Fig 1, all the main effects were found to be significant, indicating each individual factor has a significant impact on the outcome. Interaction effects being significant, except for the AB interaction, indicate that the combined influence of most pairs of factors had a statistically significant impact, but the combination of factors A and B (i.e. SFR*TEMP) didn't show a significant joint effect on the outcome.

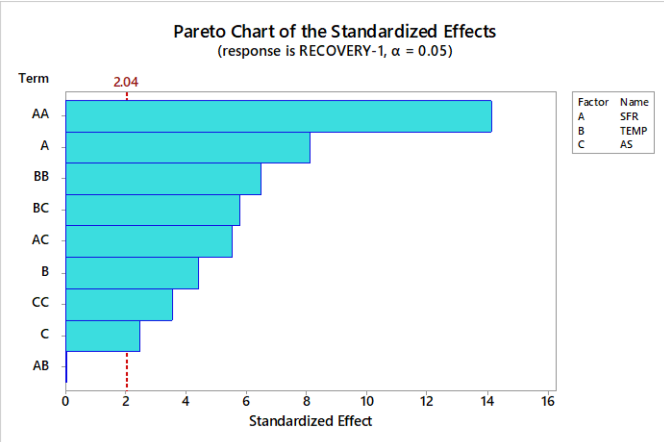

Fig 1. Pareto Chart

Fig 2 shows the main effect plot of the parameter on the response variable indicating nonlinear relationship. Each curve on the plot represents the relationship between a specific parameter and the response. A nonlinear relationship between variables means that the change in one variable doesn't result in a proportional change in the other variable. In other words, the relationship between the predictor (parameter) and the response variable isn't a straight line or doesn't follow a linear pattern. This nonlinearity might suggest that the impact of the parameter(s) on the response is more complex, possibly involving interactions, thresholds, or other nonlinear patterns.

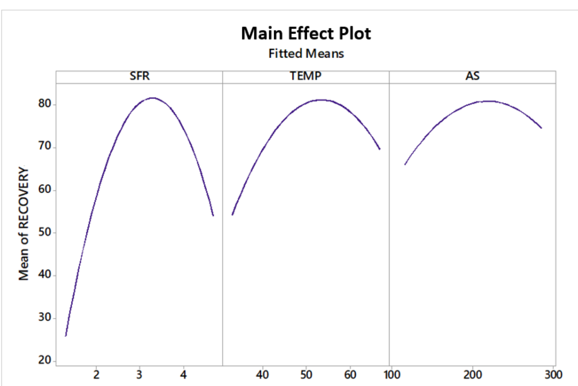

Fig 2. Main Effect Plot

3D Surface Plots are graphical representations used in experimental design and statistical analysis to visualize the relationship between two independent variables (process parameters) and one dependent variable (response). The plot is three-dimensional, with the vertical (Z) axis representing the response variable (in this case, Percent Recovery), the two horizontal (X and Y) axes represent the uncoded levels of two independent process parameters (Fig 3). By visualizing the response surface in three dimensions, the nature of interactions between these two parameters on the outcome is observed along with the optimal value of the response.

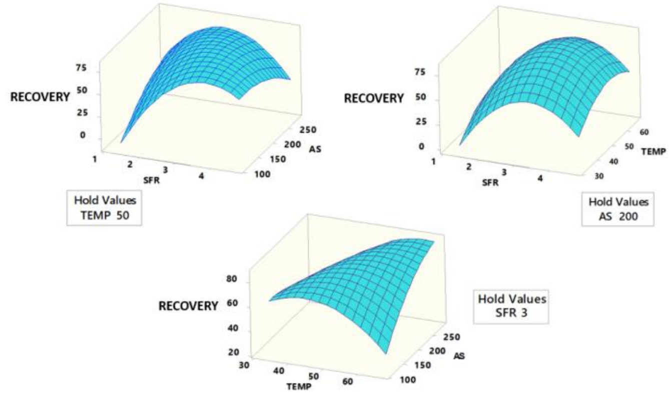

Fig 3. Surface Plot

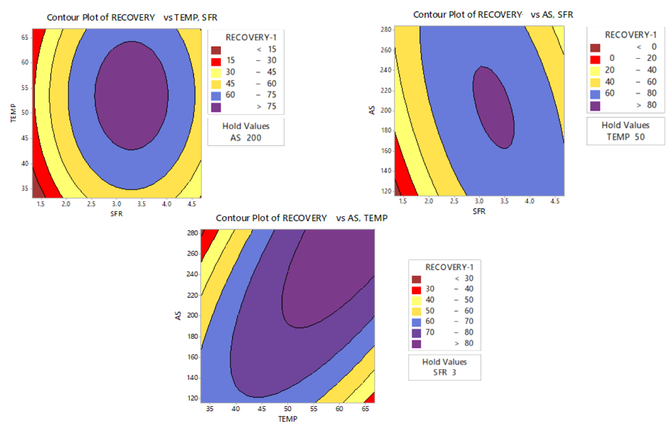

Fig 4. Contour Plot

In statistical analysis, a contour plot is a graphical representation used to display the relationship between two continuous variables. It shows how a response variable (such as recovery in this case) changes concerning two predictor variables (process parameters) by displaying contours or lines that connect points of equal value for the response variable. From the contour plot (Fig 4), it can be observed that more than 75% of the recovery is at the mid-level of process parameters. Contour plot suggests that a significant portion (more than three-fourths) of the highest recovery values is concentrated around the mid-level values of the process parameters. This finding implies that specific combinations of these process parameters, likely those at or near the mid-level, contribute to higher recovery rates compared to other combinations.

Model Validation

The optimum settings from RSM optimizer (obtained using MINITAB software) are presented in Fig 5. After obtaining these optimal settings, the validity of the model was assessed by conducting an experiment using these recommended settings. This experiment aimed to verify whether the predicted optimal settings from the RSM analysis indeed resulted in the expected outcome (e.g., maximum recovery).

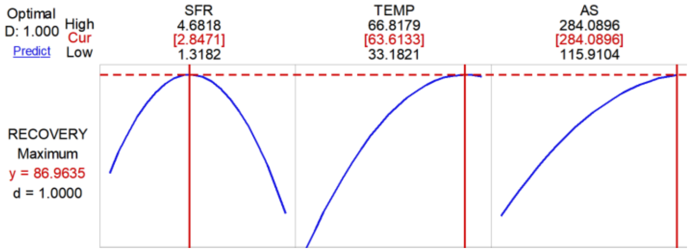

Fig 5. RSM Optimizer

Table V. Optimal Settings of Process Parameters

Factor	Setting	Model predicted value	Experimental value
SFR	2.85	86.96	85.25
Temp	63.61		
AS (rpm)	284.10		

With the Parameter settings mentioned in Table V, experiments were performed in triplicates. The measured percent recovery of 85.25 closely aligns with the predicted value of 86.96 obtained from the regression model, indicating good agreement between the two. 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 VI. For each question, 0-10 scale was used, 0 being the lowest score and 10 being the highest. 42 students participated in the study.

Table VI. Questionnaire for student Feedback

Q. No	Question
1	Training on Standard Operating Procedures (SOP) and Good Laboratory Practices (GLP) was effective and helpful in implementation Open ended experiment
2	Hands on training on Design of Experiments (DoE) was helpful in proper Implementation & successful execution of open-ended experiment
3	Implementation of the Design of Experiment (DoE) tool enhanced the ability to design & conduct experiments, analyze & interpret the results, and synthesis of the information to provide valid conclusions
4	This activity was helpful in strengthening the domain knowledge and in deeper understanding of important concepts of Solid-Liquid Extraction (Leaching) operation
5	The activity enhanced effective report writing and presentation skills
6	Able to Demonstrate knowledge and understanding of the core principles and apply these to one's own work, as a member and/or leader in a team, to manage projects
7	Are you satisfied with the overall methodology and content delivery of activity?

4. Results

Executing an open-ended experiment through the Design of Experiments (DoE) methodology in the Unit Operations Laboratory played a pivotal role in enhancing proficiency in the domain knowledge, utilization of statistical software, analytical capabilities, interpretation ability, and the capability to take informed decisions. The assessment of attainment of Program Outcomes (POs) by aligning with competencies and Performance Indicators (PIs) is conducted. The attainment of various PIs, on a scale from 1 to 10, was measured and is illustrated in Figure 6.

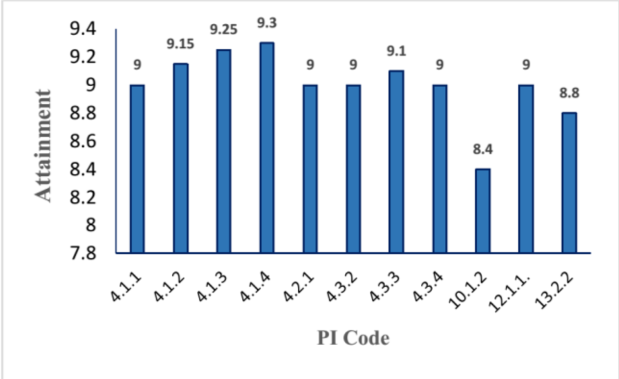

Fig 6. Attainment of program outcomes and performance indicators

This indicates that the utilization of an open-ended experiment with the DoE approach elevated proficiencies like the capability to design, execute, analyze, and comprehend the experimental results. The attainment of PO-12 was commendable, demonstrating students' capacity to participate in self-directed and lifelong learning within the framework of technological advancements.

Nonetheless, there exists potential for enhancement in the area of Communication (PO-10), which encompasses aspects such as report composition, presentation skill. There is scope for improvement with respect to strict adherence to SOP- Standard Operating Procedures and GLP- Good Laboratory Practices (PSO-13).

The feedback of students regarding the execution of Open-Ended Experiment was analyzed. The results are shown in Fig 7. The students' feedback revealed that the framework of Open-Ended experiment was very helpful in enhancing the domain knowledge and deeper understanding of important core concepts. The students expressed satisfaction in terms of having acquired the ability to design, conduct experiments, analyze & interpret the results to draw accurate conclusions. Students expressed their satisfaction regarding the overall approach adopted and the manner in which the content delivered in the process of implementation of open-ended experiment

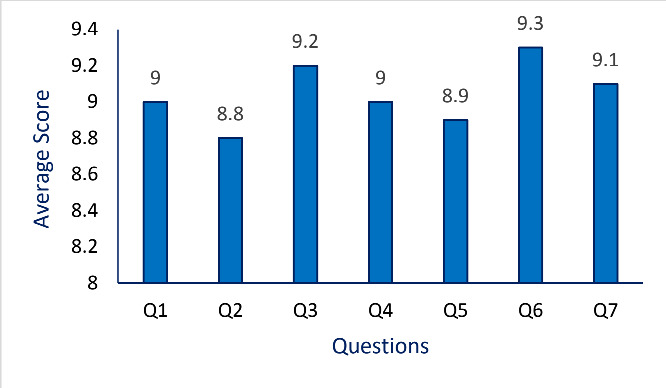

Fig 7. Students' feedback

However, there exists an opportunity for improvement in the area of hands-on training on DoE, as well as report writing & presentation skills.

5. CONCLUSIONS

Based on the findings of the current study, it can be inferred that the introduction of open-ended experiments in Unit Operations laboratory course offers an independent, innovative, and creative platform to execute experiments. The application of Statistical Design of Experiments facilitates students in effective planning and execution of experiments. By analyzing data and subsequently interpreting results, students gain a deeper understanding of Unit Operations laboratory concepts through experiential learning, leading to improved attainment of PO-4. Open-ended experiments enable reliable assessments by utilizing rubrics for evaluating lab reports, teamwork, and practical skills. According to student feedback, the use of Design of Experiments (DoE) enhances domain-specific skill sets and proficiency in using statistical software. Nevertheless, there is a scope for improvement in area of written and oral communication (PO-10).

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