Response to the "Evaluation report of Ph.D. Thesis"

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Sr. No.	Comment	Response
1. Comments from Dr. Umesh Kumar Khare		
1a. General comments		
1.	**Organisation** – Thesis presented on electrochemical and peroxymonosulfate based processes for pharmaceutical compounds is well organised. Background and literature review presented showed comprehensive review of the research till date. Experimental design and methods are clear and result and discussion section is presented in logical format.	We are grateful to the Examiner for such encouraging remarks.
2.	**Quality of work –** Quality of work is very good and involved with new ideas. Thesis is comparable with other universities of repute.	We are grateful to the Examiner for such encouraging remarks.
3.	**Strong points -** Quenching study to find best quenching agent is appreciable. It's good to see that researcher has done LC-MS analysis for IPs. Study performed for finding higher contribution of sulphate radical compared to hydroxyl radical in EC/PMS process is excellent.	We are grateful to the Examiner for such encouraging remarks.
4.	**Weak points –** i. Gap in the literature studied by researcher is not clearly mentioned and how to fill up that gap in his research work is not included in the need of study or in aim and objectives of the study. ii. Lack of attention given towards numbering table of contents, list of figures and list of tables in the thesis as it was found that almost all are numbered wrongly. In results and discussion sections lots of table and figure numbers are shown incorrectly. iii. Some of the abbreviations used in thesis are not included in abbreviation list. iv. At several places space gap between full stop and new sentence is missing.	We accept all the points and we have made all necessary corrections. i. Gap in literature studied is mentioned at the end of sub topics 2.1 and 2.2 individually. And how this study can fill up this Gap is now included in the sub topic 1.5, i.e. need of the study. ii. Numbering is thoroughly checked and corrected at all places. iii. Abbreviation list is corrected and all abbreviations are now included. iv. Space between full stop and new sentence is corrected at all places.

5.	***Looking to technical contents of thesis I have no hesitation to recommend for the award of Ph.D. degree.***	We sincerely thank the examiner.
1b. Comments to be answered at the time of Viva		
1.	Why only one initial concentration of 10 mg/L (DCF and IBU) is used in whole study, though in discussion it was said that concentration of compound is also one of the factor affecting degradation time and rate?	The PhCs studied here are usually present in a trace amount ranging from ng/L to mg/L in natural waters. To simulate this and also keeping the ease of analysis (using HPLC) in mind, 10 mg/L was decided for both DCF and IBU. The results obtained in our study can be applied to lower concentrations of PhCs too, which is normally found in natural waters. Also, effect of various concentration is studied as per the literature review (El-Ghenymy et al., 2013; Tan et al., 2014; Y. Wang et al., 2016) and authors reported that higher initial concentration adversely affect the degradation efficiency.
2.	In the EO study for S:C ratio, TDS is maintained 1000 mg/L synthetically in all experiments but as per ROC characteristics TDS was 3000 mg/L in ROC. Why it was not kept same? Please justify?	The study pertaining to S:C ratio was performed using synthetic ROC prepared by addition of NaCl and Na_2SO_4 in distilled water to achieve a required S:C ratio. The main goal of this study was to evaluate the effect of relative abundance of sulfate and chloride ions on the removal of IBU using EC/PMS rather than the total concentration of sulfate and chloride ions. The optimum ratio of S:C derived from above study was applied to actual ROC (by adding sodium sulfate) and synthetic ROC having TDS = 3000 mg/L. % IBU removal was the maximum in both the experiments wherein the optimum S:C ratio was used.
3.	What is theoretical aspect of using MMO other than Graphite for comparison? How, the mol ratio of Ru:Sn:Sb was decided to be used in MMO in the study ? Explain it.	MMO is a dimensionally stable anode, but graphite is not, especially at higher currents. Also, coated oxides may act as catalysts and can help in degradation. Molar ratio of Ru:Sn:Sb was adopted from the previous study "Electrochemical destruction of RB5 on Ti/PtOx–RuO2–SnO2–Sb2O5 electrodes: a comparison of two methods for electrode preparation" (Soni et al., 2020).

4.	In the Figure 6, maximum RCS concentration is shown as 453 mg/L. Please explain the method of its determination which can be included in the chapter of material and method.	It was mentioned in first paragraph of topic 3.3 that iodometric method (Palma-Goyes et al., 2016; H. Wang et al., 2019) was used and it is now elaborated – " Two drops of acetic acid were added in 5 mL volume of 50 times diluted aliquots to bring down pH 3 to 4. Two drops of KI solution (40000 ppm) were added to develop yellow color which was measured at 350 nm using UV-visible spectrophotometer (Brand: Shimadzu, Model: UV1800, Spectral Bandwidth: 1 nm)".
5.	i. In RSM analysis- Table 6 – Run 7-11 all are having pH-7.5, PMS- 500 mg/L and CD-2.5 mA/cm^2 though variations are shown in response 1 and 2. ii. Higher value of CD for pH-7.5, PMS- 500 mg/L is not shown. Please discuss and justify.	i. Firstly, in RSM analysis, for Run 7 to 11, standard deviation is 0.5 and coefficient of variation is 0.5% for response 1, which is reasonable and the variation was because of measurement uncertainty. For response 2, we have considered the reaction rate constants for trendline passing from origin and not the actual trend, this was the reason of high coefficient of variation 9.9%. After the remark made by reviewer, we have considered the original trend and reaction rate constants accordingly. Standard deviation is 0.003 and coefficient of variation is 6%, which is reasonable. Also, this variation do not affect the overall developed model. ii. After preliminary experiments, the combination pH-7.7, PMS-500 mg/L, CD-2.5 mA/cm^2 was found as an optimum point for % IBU removal. BBD design was adopted to develop mathematical model, so that the effect of these parameters is explained better. Moreover, design of Box Behnken method was followed and 17 experiments were decided this way to develop a mathematical model. These 17 experiments suggested by Box Behnken Method did not include higher CD for pH – 7.5 and PMS – 500 mg/L.
6.	As per literature in Table 1 and 2 for DCF and IBU removal EO and PMS activation methods are used by researchers but you have not used both the methods for both PhCs –justify your decision.	As per the first objective of the study, it was decided to explore different processes for different PhCs and try the combination (as we did in form of EC/PMS). While carrying out the experiments, we encountered certain issues which were relevant and important for the study and were not

		explored before e.g. effect of quenching, S:C ratio, RSM for EC/PMS, reaction in continuous flow mode, LC MS analysis, etc. Therefore, we chose to carry out two exclusive studies. Developed methodologies for both the studies can also be applied interchangeably to IBU and DCF.
7.	What is logic behind choosing very odd increments in PMS (100, 500 and 900) and CD (.525, 2.5 and 4.475) in the study of effect of pH in IBU removal?	After preliminary experiments, the combination pH-7.7, PMS-500 mg/L, CD-2.5 mA/cm2 was found as an optimum point for % IBU removal. It was the requirement of BBD method that points must be equidistance. For PMS we could not choose 0, 500, and 1000; as change from 0 to 500 is drastic and it would overshadow the actual effect. That is why 100, 500 and 900 was selected. Also, it was found from preliminary experiments that at PMS<100 mg/L, the removal of IBU was not significant. On the other hand at PMS>700 mg/L, there was not uch improvement in IBU removal beyond that obtained at 500 mg/L. Similarly, for current density, 0.525, 2.5, and 4.475 was chosen because it showed significant change from 0.525 to 2.5 and 2.5 to 4.475.
8.	In 4.6 it is said that treated water can be reused to irrigation purpose but in conclusions section it is mentioned that it was presumed so please make clear that quality of treated water is safe for irrigation from sodium contents and SAR point of view.	It is accepted that sodium contents and SAR must be checked for reuse application such as irrigation. Moreover, ROC having higher TDS than 2100 mg/L must not be reused for irrigation purpose. Therefore, it is corrected in conclusion as below: “Hence, it was concluded that ROC was free from toxic end products after EO treatment for DCF removal”. Also, correction was made in Results and discussion section – “Similarly, for phytotoxicity, initial value was 3% which marginally increased to 9% after 120 min of treatment. It has been reported that any marginal increase in the phytotoxicity after the treatment may not have substantial adverse effect on plant growth (Santos et al., 2020; Vijayakumar et al., 2021). It is important to note that ROC can be used for irrigation only if the TDS limit and

		Sodium Adsorption Ratio for the effluent are met."
1c. Chapter wise evaluation- Minor comments		
Abstract		
1.	Abstract first page-second last line- limited reuse potential cause of high salt content – It should be limited reuse potential because of high salt content.	Corrected
2.	Abstract third page-14th line- both %IBU removal – It should be both % IBU removal	Corrected
Chapter 1		
1.	Page 1 line 5 - space between word end and bracket	Corrected
2.	Page 1 line 9 – space between coma and new word	Corrected
3.	Page 2 line last – space between full stop and new word	Corrected
4.	Page 3 line 2 – space between full stop and new word	Corrected
5.	Page 3 line 10 – space between full stop and new word	Corrected
6.	Page 3-line 21- PhC remain as it is cause of its strong persistent – It should be PhC remain as it is because of its strong persistent	Corrected
7.	Page 4 line 8 – space between full stop and new word	Corrected
8.	Page 4 line 15 – space between full stop and new word	Corrected
9.	Page 6 ECO and EO both are used in thesis several times but in abbreviation list EO is listed for electrochemical oxidation. ECO is used for electrochemical oxidation thrice on this page and on other pages- 6, 32, 36, 42, 56 and 57 also while EO is used on other places.	ECO is replaced with EO on pages 6, 32, 36, 42, 56, 57. EO is the only abbreviation for Electrochemical Oxidation for this thesis.
10.	Page 6, 1.3- line 15 and 22 - space required between OH* and next word	Corrected
11.	Page 7 line 2 – reported pole apart results (Y. Yang, 2020) should be checked	Corrected
12.	Page 7, 1.4 - line 5 - space between word end and bracket	Corrected
13.	Page 7, 1.4- line 9 - space between full stop and new word	Corrected
14.	Page 7, 1.4- last line - space between full stop and new word	Corrected
15.	Page 9, 1.6 last objective To identify end products. It should be To identify Intermediate products	Corrected
Chapter 2		
1.	Page 10, 2.1 - line 10 - space between word end and bracket	Corrected

2.	Page 20, 3rd line - space between full stop and new word	Corrected
3.	Page 20, 5th line –line repeated as in abstract with same mistake	Corrected
4.	Page 20, 7th line – PPCPs – should be included in abbreviation list	Corrected
5.	Page 20, line 11 - space between word end and bracket	Corrected
6.	Page 21, 2.2 - line 9 – extended life – around 30 to 40 μs should be checked	Corrected
7.	Page 21, 2.2 - line 17 – activation thru transition	Corrected
8.	Page 29, Lower portion of first paragraph is almost repeated as on page no 8	Corrected on page no. 8 (now page 7, last paragraph)
	Chapter 3	
1.	Page 31, lots of space left on top as well as font size for subscripts are different for different formula	Corrected
2.	Page 32, 3.2,line 3 - space between full stop and new word	Corrected
3.	Page 33, line 18 – 20 min, and 15 min respectively, should be 20 min and 15 min RT respectively	Corrected
4.	Page 33, line 21 – Rector should be Reactor	Corrected
5.	Page 34, Schematic view of EO of DCF can be included as Figure 2 is for EC/PMS of IBU	Schematic view of EO of DCF is now included as Figure 2. EC/PMS of IBU is shown in Figure 3 and 4.
6.	Page 38, 3.4, line 1 – space required between 10 mg/Land	Corrected
7.	Page 39, equation 1 for phytotoxicity need to be written properly instead of copy and paste	Corrected
8.	Page 39, 3.6, line 6 – need to be checked and explained	There was a cross reference error, which is corrected as Figure 6.
	Chapter 4	
1.	Page 41, 4.1, line 3 – remove; from end of OCl	Corrected
2.	Page 47, 6th line – use Appendix number instead of writing refer to supplementary information	Corrected
3.	Page 49, 4.3, line 8 – insert space behind applied density	Corrected
4.	Page 49, 4.3, line 16 - space between full stop and new word	Corrected
5.	Page 52, Figure 10 (c) S:C ratio need to be checked	Corrected
6.	Page 54, line 13 - space between full stop and new word	Corrected
7.	Page 57, Figure 14 – use (a) and (b) also in (b) –Y-axis Total in place of Totao	Corrected
8.	Page 58, 4.6, line 6 – mentioned Table 2 is not for phytotoxicity results	Corrected

9.	Page 58, 4.6, last line – mentioned Figure 14 is not for phytotoxicity results	Corrected
10.	From Page 60 to Page 76 in results and discussion, almost all numbers for Figure are incorrect	Corrected
11.	Page 61, line 20 – use because in place of cause	Corrected
12.	Page 68, line 8 – use because in place of cause	Corrected
13.	Page 73, Use of Figure S1, S2, S3 and S4 in discussion are not marked like this in thesis anywhere	Figure S1, S2, S3, and S4 were wrongly mentioned for Appendix F, G, H, and I. Paragraphs on page 73 is corrected for these with correct references.
14.	Page 74, 4.10, line 3 – Rector should be Reactor	Corrected
15.	Page 77, Font size of numeric used in LC-MS spectrums is different	Corrected. Complete uniformity is not there because some data are given with different scale.
Chapter 5		
1.	Chapter name should be conclusions instead of conclusion	Corrected
2.	Most of the conclusions are written with lots of discussion statements. So much of discussion could have been avoided by the researcher as it was already written in results and discussion section	Conclusion are re-written and discussion is avoided.
3.	Page 81, second last statement, 1st line – space between % and IBU as used at other places	Corrected
Appendices		
1.	In appendix G, H and I [PMS]0 and [IBU]0 should be with 0 in subscriptions	Corrected

2. Comments from Dr. Sushil Kanel

Sr. No.	Comment	Response
2a. General comments		
1.	I am commenting on the thesis of Katha Hirpara (advisor Dr. Upendra Patel). The work is excellent and essential to the scientific community. Katha has done a fantastic job under the guidance of Dr. Patel; I approve the thesis with my minor comments.	We are grateful to the Examiner for such encouraging remarks.
2b. Specific comments		
1.	Katha needs to explain the degradation of pharmaceutical compounds (PhCs) used in this research based on recent literatures.	We have used several recent references while discussing the results of PhCs degraded in our study. Few additional recent references are also added now in *Literature review & Results and discussion* sections.
2.	In the abstract, I suggest using our environment instead of "leads to the accumulation of such compounds in surface water and groundwater." The contaminant will also enter to soil and groundwater aquifer through the water.	We accepted the suggestion and "our environment" is used in place of "surface water and groundwater".
3.	In the abstract, are all pharmaceutical compounds (PhCs) toxic to ng/L level? What about mg/L? Please check and make sure you are reporting correctly.	It has been reported that a wide variety of PhCs including DCF and IBU, in ng/L to mg/L are found to cause inhibitory effect on several test organisms such as V. fischeri and S. capricornium alga (Balakrishna et al., 2017; Khan et al., 2020; J. Zhang et al., 2021).
4.	In the abstract, rewrite "to degrade these compounds to Mineralization."	Corrected
5.	In the abstract, write AOP such as ozonation.	Corrected
6.	In the abstract, mention the abbreviation used for the first time, e.g., write a complete form of HPL and LC-MS.	Corrected
7.	Is the abbreviation of intermediate as IP scientifically used in the community, or are you making one yourself? Better to write in the full form if not used in the scientific community. Similar issued with CD and others.	"IPs" is used for "Intermediate Products" in the community (Cheng et al., 2021; Filz et al., 2020; Levin et al., 2012; Y. Zhang et al., 2019). Same with the "CD", it is being used in community (Abdulhadi et al., 2019; Belhadj et al., 2018; Joy et al., 1999). However, now

		we have corrected IPs and CD all over the draft where it is coming in the text.
8.	Introduction 1.1 Background write as millions of people instead of millions only.	Corrected
9.	In section Introduction 1.1 Background, be consistent in writing PhCs as appropriate all over the place.	Corrected at all places.
10.	In 1.2, as AOP is written in complete form no need to write full form use AOP.	Corrected as "AOP"
11.	On page 5 literature review cited by Kanakaraju et al., 2018 little old adds one more recent review paper as a reference.	Recent references are added in Literature review section.
12.	EO or ECO? Be consistent all over the thesis.	The draft is reviewed and now "EO" is used consistently for electrochemical oxidation, all over the thesis.
13.	In abbreviation, check S: C [Sulfate]/[Chloride]mass ratio, S; C or Cl?	It is "S:C" for the ease of writing.
14.	Add CD (page 81) and others mentioned in the draft in the Abbreviation section.	All the terms are included in abbreviation list.
15.	Add 2021 and 2022 papers in Table 1 and Table 2.	Two relevant papers are added in each table. Total four recent papers are added.
16.	Fig7b, explain why the red and blue lines are missing or hiding on the black line!!	Red and Blue lines are not hiding on the black line. They are missing because of the reason written in section 4.2, page 42: The reason behind this is the ability of sulfite to reduce halogenated compounds and azo-aromatic compounds (Diana et al., 2019; How et al., 2017). IPs are possibly reduced by sulfite and that is why they are not detected in the sample. Here, the concentration of sulfite is much higher than DCF and IPs concentration, this could also be the reason. This false negative determination of IPs can mislead the investigation and researchers may draw the wrong conclusions. Kristiana et al., 2014 and Yang & Zhang, 2016 also reported in their studies that sulfite cannot be used while studying the halogenated organic compounds. Kristiana et al., 2014 showed that sodium arsenite is the alternative to sodium sulfite while working with halogenated organic products.
17.	Page 55 adds what was degraded, for example, Three different electrodes – i. Ti/Ru-Sn-Sb-Ox, ii. Ti/Ru-Ir-Ox, and iii. Graphite was used as an anode keeping other reaction parameters the same, to evaluate the	This is corrected as "Effect of electrode material on EO of DCF"- in the heading and the paragraph.

	effect of electrode materials on the degradation of DCF.	
18.	Page 60 mentions that "As shown in Figure 15," but figure 16 was referred-please check carefully here and all over the draft.	Reference to the Figures are checked and corrected all over the thesis.

References:

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