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Evaluation of Commercially Available Precursors for Obscuring Wet Industrial Irradiator Facility Pools to Delay Adversarial Source Removal. Part 2

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ABSTRACT

The behavior of commercially available potential obscurants for cobalt-60 (^{60}Co) wet-source storage industrial irradiator facilities (IRFs) were further evaluated for corrosive behavior of Nordion C-188 pencil stubs and obscurant properties under radiation exposure (^{60}Co). The potential obscurants studied included: titania aqueous dispersions (TAD – water soluble white paint), Chlorazol Black (CBOD – Chlorazol Black organic dye), powdered milk (COW – calcium obscurant in water), diatomaceous earth (DEA – diatomaceous earth additive), and rhodamine 6G (R6G). For corrosion efforts, stubs from an inert C-188 pencil-source rod were soaked in obscurant solutions and visually inspected. For radiation stability, obscurant samples were exposed to ^{60}Co radiation sources at 207 rad/s. The results from these studies reveal: the obscurants had no impact on the surrogate samples and may assist in terms of corrosion resistance; materials that did not rely on organic compounds to provide obscurance performed the best, as the organic compounds decomposed too rapidly in the high radiation environment, whereas the materials survived.

ACKNOWLEDGEMENTS

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ACRONYMS AND DEFINITIONS

Abbreviation	Definition
BRUCE	Basic Research for Underwater Clarity Evaluation
C	carbon
CBOD	Chlorazol Black organic dye
Ci	curie
cm	centimeter
Co	cobalt
COW	calcium obscurant in water
DEA	diatomaceous earth additive
DI	de-ionized water
g	gram
GIF	gamma irradiation facility
h	hour
H_2O	hydrogen dioxide
HCl	hydrochloric acid
IRF	industrial irradiator facility
min	minute
mL	milliliter
mM	millimolar
NNSA	National Nuclear Security Administration
NTU	Nephelometric Turbidity Unit
ORS	Office of Radiological Security
R6G	rhodamine 6G
s	second
TAD	titania aqueous dispersions
TiO_2	titanium dioxide
μS	micro Siemen

1. INTRODUCTION

In support of the National Nuclear Security Administration's (NNSA's) Office of Radiological Security (ORS) program, this project aimed to develop methods that would slow an assailant's intent on extracting radioactive material from industrial irradiator facilities (IRFs). A schematic of a typical 'wet-IRF' is shown in Figure 1.[1] Previously, we reported on the utility of a series of potential commercially–available chemicals and materials evaluated as potential obscurants for water pools used to store and shield IRF ^{60}Co radiation sources, including: titania aqueous dispersions (TAD – cheap water soluble white paint), Chlorizol Black (CBOD – Chlorazol Black organic dye), powdered milk (COW – calcium obscurant in water), diatomaceous earth (DEA – diatomaceous earth additive), and rhodamine 6G (R6G). Each of these obscurants was independently evaluated in a beaker and a simulated IRF tank termed Basic Research for Underwater Clarity Evaluation (BRUCE).[2] Both TAD and CBOD samples proved to be effective at rapidly obscuring the water in the tank, maintaining dispersion, and falling within the predetermined IRF safety parameters. Through the addition of anti-TAD (calcium and aluminum salt mixtures), TAD and CBOD were successfully precipitated from BRUCE and removed by vacuum or filtration.

[1] Chmielewski, A. G., Gamma Irradiators for Radiation Processing (Report number INIS-XA--862). Agency, I. A. E., Ed. Vienna (Austria), 2006; pp 1-46.

[2] Boyle, T. J.; Reuel, P. C.; Reinholtz, W. D.; Romero, M. P.; Thompson, A. D.; Gilbert, L. J.; Kuca, M.; Cook, A. W., SAND2019-7727: Evaluation of Commercially Available Precursors for Obscuring Wet Industrial Irradiator Facility Pools to Delay Adversarial Source Removal. 2019.

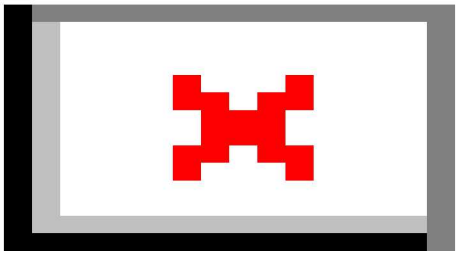


Figure 1. Schematic diagram of a typical wet IRF

While new obscurants are being developed and evaluated, further studies on these obscurants for corrosion resistance and radiation stability were undertaken. The Corrosion Studies section discusses the corrosive nature of these obscurants on the stability of the C-188 stainless-steel rods that encase ^{60}Co sources. This was explored by 'soaking' experiments and through optical evaluation. The Radiation Studies section presents the impact that exposure of obscurant solutions to ^{60}Co had on the solution properties. Solution exposure for the study took place at the Gamma Irradiation Facility (GIF) at Sandia.

2. CORROSION STUDIES

Obscurants used in IRF pools must not adversely impact the integrity of the stainless steel encapsulated ^{60}Co sources. Therefore, it was necessary to understand how the different obscurants might interact with the pencil source stainless-steel encasements. Encasement samples (or slugs), were cut from the original surrogate pencil source (using a hacksaw) and then placed into a solution over an extended period of time. Two sets of reactions were run to study this. The first involved the following solutions: (i) de-ionized (DI) water, (ii) concentrated hydrochloric acid (HCl), and (iii) TAD. These were selected to show a baseline, a corrosive environment, and one of the more successful obscurants. After this testing, the same three samples were repeated and all of the other obscurants (COW, CBOD, and DEA) were also studied. The details of each are discussed below.

Corrosion Experimental. All samples were prepared on a benchtop as pictured in Figure 2. An inert C-188 pencil source rod, which consisted of nonradioactive ^{59}Co in place of the radioactive ^{60}Co, was supplied by Nordion. Sections or slugs were cut from this pencil with a tube cutter and used without additional manipulation. Each slug was characterized by optical imaging using a VHX-5000 microscope.

Each slug was added to a beaker and soaked for pre-set times: (i) DI water (300 mL); (ii) (conc) HCl (150 mL H_2O/150 mL ~12 M HCl); (iii) TAD (0.59 g, 300 mL H_2O); (iv) CBOD (0.059 g, 300 mL H_2O); (v) COW (0.074 g, 300 mL H_2O); and DEA (0.118 g, 300 mL H_2O). These values represent the obscurant at a concentration consistent with that used in the BRUCE test environment. After the obscurant was stirred to disperse it through the water, a slug was added. Optical images were collected (at 5, 30, and 60 minutes) by removing the sample, rinsing it off, and wiping it dry followed by image collection. These images were also collected at 21, 46, 147, and 188 hours in the same manner.

Corrosion Results and Discussion. Three solutions were initially investigated: (i) DI water, (ii) (conc) HCl, and (iii) TAD; and the original slugs are shown in Figure 2, (a). Aged samples are shown in Figure 2, (b) through (d).

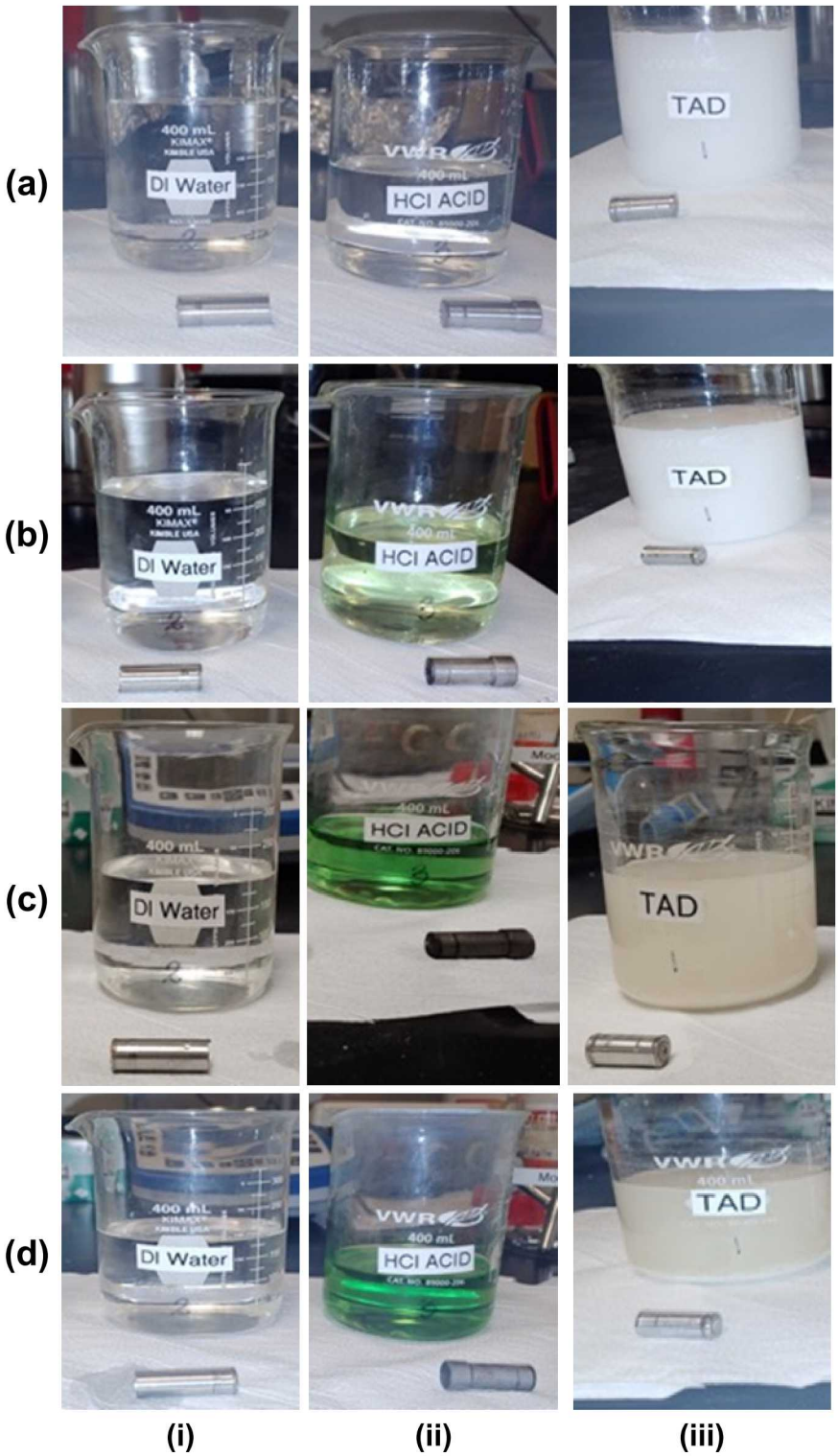


Figure 2. Picture of the various solutions (i) H_2O, (ii) HCl, and (iii) TAD and the slugs used for corrosion study at time = (a) 0 h, (b) 46 h, (c) 178 h, (d) 196 h.

Water. It was expected that there would be no impact on the slug upon soaking in water, as this is the surrogate used in the IRF setup. However, as can be clearly observed in the Figure 3, A Post photo, there is considerable rust present after the relatively short soak of 46 hours. Corrosion was seen in the areas that had been cut or scratched by the steel tube cutter. It is common for stainless steels to develop this corrosion in areas that may have contaminants from the cutting process. The pristine areas of the slug did not show signs of corrosion.

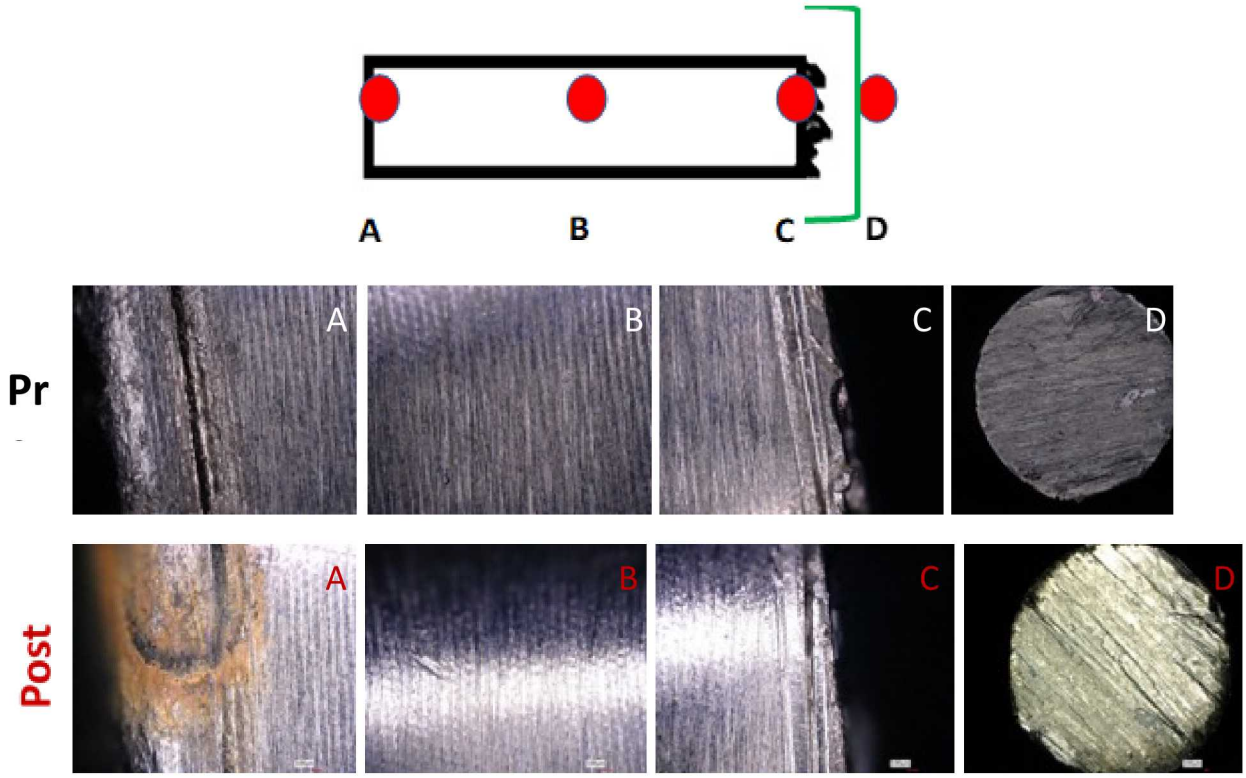


Figure 3. Schematic and resultant optical images at (i) pre (0 h) and (ii) post (46 h) in H_2O.

HCl Solution. The next solution investigated was concentrated HCl. This solution was investigated to have chemically induced corrosion in order to characterize this behavior. Not surprising, after soaking for 46 hours (Figure 4) there is significant pitting, etching, and loss of materials for the post. This occurred throughout the sample and not only where the slug had been sawed. In particular, the terminus (Figure 4, D Post) shows the removal of material associated with the soak. As can be observed in Figure 2, the solution turned green indicative of iron, molybdenum and nickel being dissolved from the stainless steel, which was confirmed by X-ray fluorescence (see Appendix A).

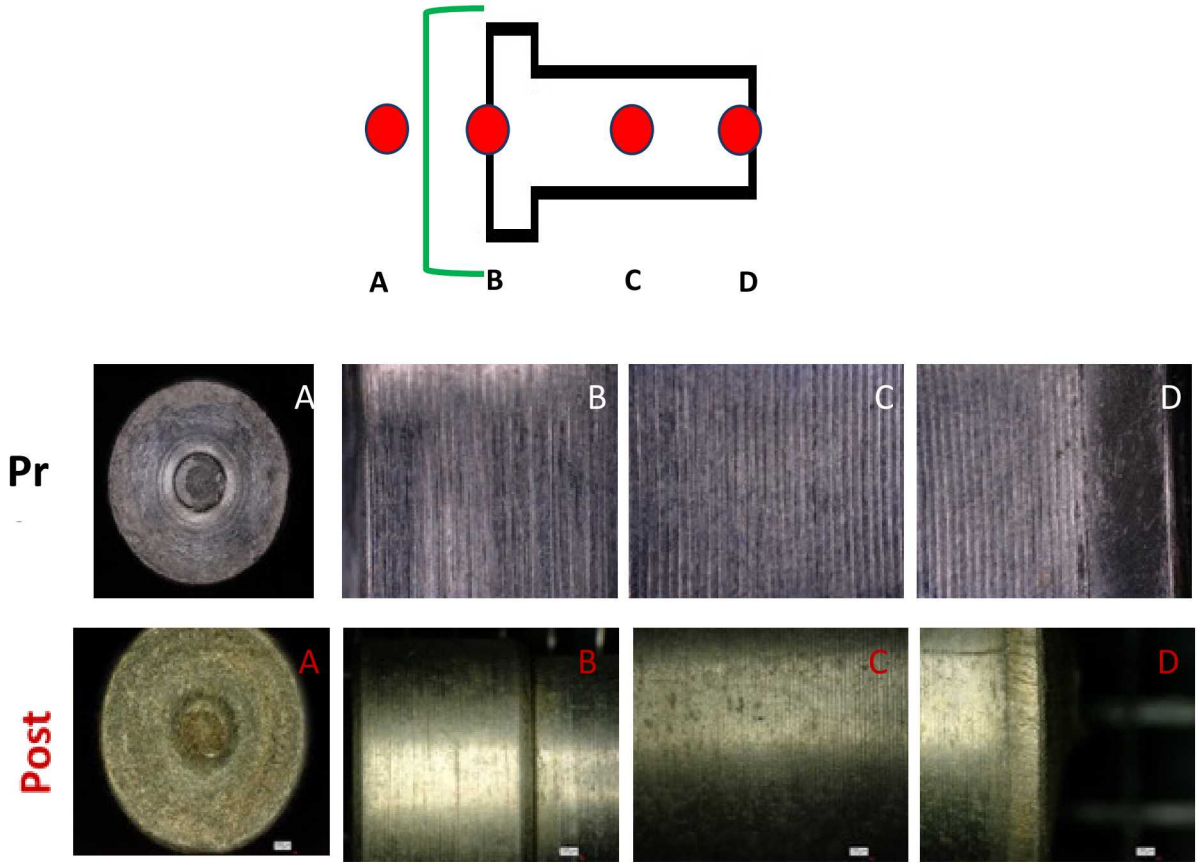


Figure 4. Schematic and resultant optical images at (i) pre (0 h) and (ii) post (46 h) in (conc) HCl.

TAD Solution. Finally, the effects of the first obscurant investigated, TAD, were explored. The results are shown in Figure 5. After 46 hours, the sample showed no decomposition (or rust). This was surprising, as the water-only sample showed rust had formed on the surface of the slugs. The surface looks clean with a possible coating of the TAD on the surface notable in Figure 5, C Post. The TAD remained suspended in solution at 46 hours, staying a bright white and providing significant obscuration without agitation (Figure 2).

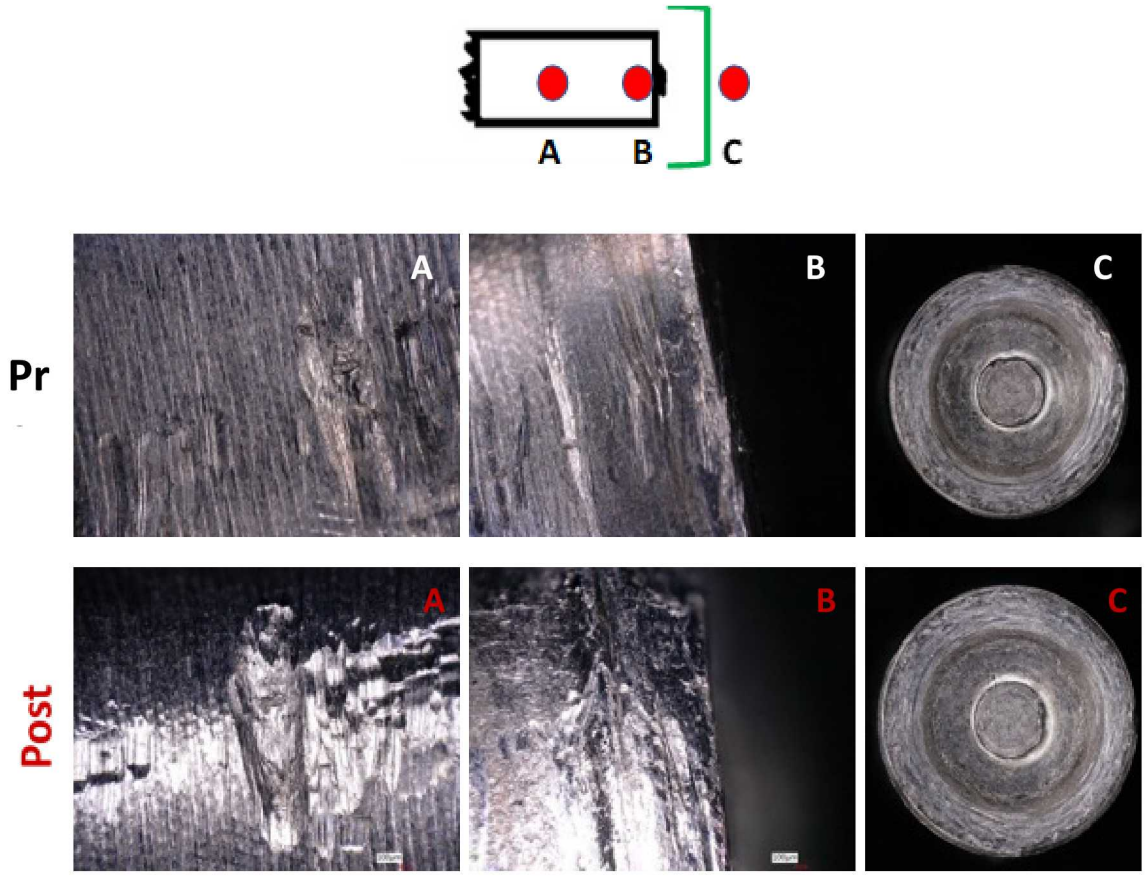
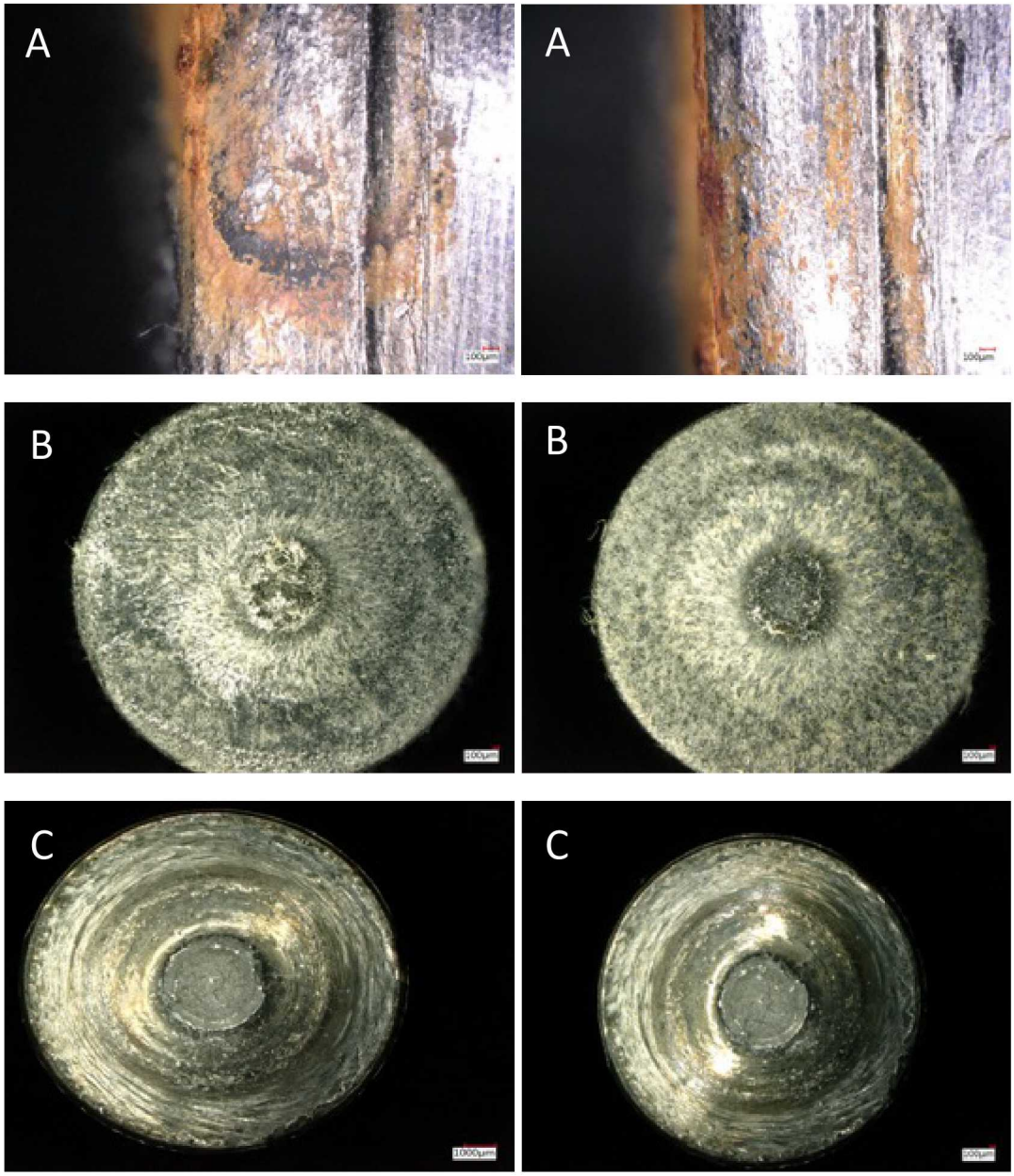


Figure 5. Schematic and resultant optical images at (i) pre (0 h) and (ii) post (46 h) in TAD

Due the surprising changes noted, additional aging studies were undertaken allowing the above solutions to age for 178 and 196 hours. As can be noted in Figure 2, for the 178 h samples, the water solution stays clear, the HCl solution gets darker green, and the TAD starts to settle with a larger precipitate noted and the solution turning a light pale yellow. Figure 6 shows the areas of the slug samples most impacted by the soaks. The water continues to rust with much more oxidation noted in the water image, Figure 6, (i)(A).



(i) (ii)

Figure 6. Select optical images of impacted areas at: (i) 178 and (ii) 196 h for (A) H_2O, (B) HCl, and (C) TAD.

For B, the distinguishing area on the point of the slug continues to increase with loss of metal to the HCl, and changes continue as the samples are further aged (196 hours, see Figure 6, (ii)). The continued pitting by simple water is surprising; whereas, the loss of metal in the HCl was fully expected. There appears to be no impact by the TAD solution, which may be due to the smaller titanium dioxide (TiO_2) particle filling in any damaged areas.

Based on the surprising rust prevention by TAD, the other obscurants were similarly studied. In addition, TAD, water, and HCl were repeated. Initial slug images and solutions are shown in Figure 7. Additionally, Figure 7 reveals the change noted over time for the slugs and solutions.

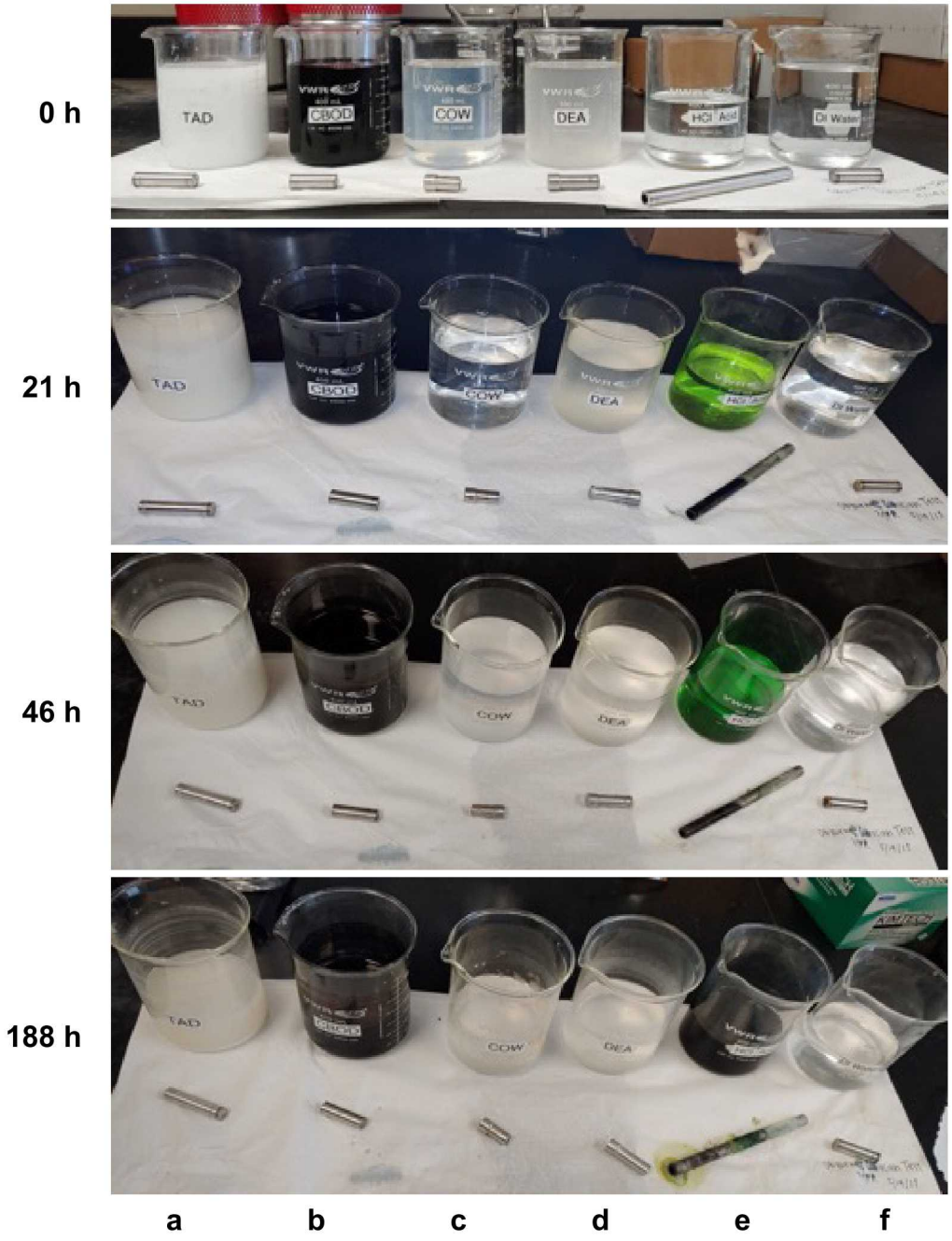


Figure 7. Select optical images of pencil slugs for: (a) TAD, (b) CBOD, (c) COW, (d) DEA, (e) HCl, and (f) DI water at hours listed.

For all of the obscurants (TAD, CBOD, COW, and DEA), similar behavior of the solution to that noted for the BRUCE tests were observed. That is, TAD and CBOD maintained their dispersion, while COW and DEA slowly settled to the bottom of the beaker. Inspection of the slugs for these were made at regular intervals through the test. For the HCl and water samples, similar decompositions as was previously noted was observed. The HCl reaction continued to decompose and color the rod (see Figure 7). Optical images were not recorded due to the lack of noted changes.

3. RADIATION STUDIES

The radiation exposure of the obscurant solutions was undertaken using high-intensity gamma-ray sources at the GIF at Sandia. For this system, a rack containing approximately 160,794 curies (Ci) of ^{60}Co pencils was used to expose the different obscurant solutions to a radiation level on the order of ~207 rad/s. All samples were prepared at similar concentrations to those used in the BRUCE testing and mixed in glass containers prior to exposure. For each experiment, the samples were brought into the irradiation chamber and placed near the edge of the pool where the source rack would be raised to be left for a total exposure at calculated times. While the exposure rate here is significantly less than what would be encountered at many IRFs, the goal was to use the results as an indicator as to whether the obscurant could withstand high-radiation areas long enough to provide adequate delay, thereby determining if the obscurant was worth further evaluation. The obscurants studied included: (a) R6G, (b) TAD, (c) CBOD, and (d) DEA. Results are discussed below.

Radiation Experimental. In 1000 mL Erlenmeyer flasks, a sample for each of the obscurants listed was prepared at a concentration consistent with that used in the original BRUCE tests (see Table 1). Samples were transferred to the GIF, where each sample was set near the irradiator pool, and a GoPro™ camera was aligned and properly shielded to record any visual changes that may occur during the irradiation. To begin, the dose rate in the location where the samples would be placed was measured using an ion chamber (206.8 rad/s). This was recorded as the baseline dose rate from which total doses were calculated.

Table 1. GIF experiment concentrations and total radiation dose to potential obscurants.

Experiment Sample	Obscurant Concentration (g/mL)	Irradiation Duration (min)	Total Dose (rads)
Baseline Dose Rate	---	4	49632
R6G - #1	11	5	62040
TAD	142	360	4466880
R6G - #2	11	30	372240
CBOD	5.4	30	372240

Radiation Results and Discussion. Table 1 lists the four obscurant samples irradiated in the GIF: (a) R6G (twice), (b) TAD, (c) CBOD, and (d) DEA. Physical appearance observations and some of the solution measurements are presented below.

(a) R6G. While R6G was not a useful obscurant in the BRUCE testing system, it was selected as the first candidate to investigate, as it reportedly scintillates upon exposure to radiation. It was expected that this system could be useful as a test-bed for other scintillators and may demonstrate an alternative route to obfuscation (i.e., bright lights). The initial R6G sample (#1) was irradiated for 5 minutes.

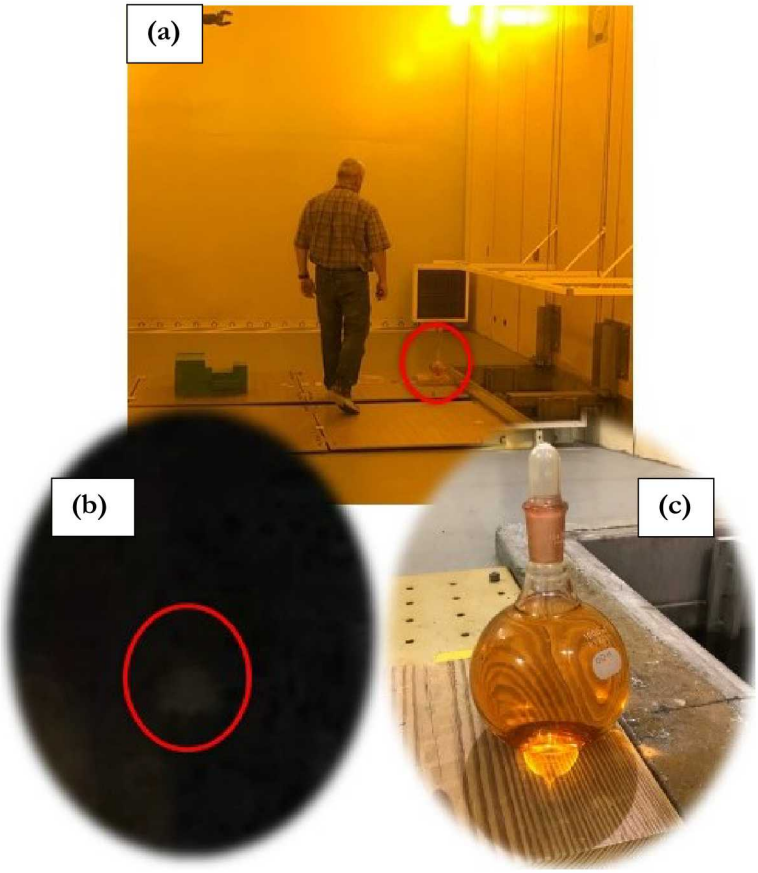


Figure 8. Images of the R6G sample (a) near edge of pool, (b) during irradiation, and (c) after irradiation (5 min).

Figure 8 (a) and (b) images are taken through the thick, mineral-oil filled glass of the irradiation chamber. The lights of the chamber were turned off during this sample irradiation so that any fluorescence could be seen. As seen in Figure 8 (b), the R6G sample glowing slightly as it is exposed to the radiation. The 0.023 mM solution continued to glow weakly for approximately 3 minutes. The concentration can be increased if a scintillation approach is of interest for a brighter response; however, the samples do decompose rather quickly, and higher concentrations would impact the water quality negatively. The same sample (R6G #1) was kept in the irradiation chamber for subsequent tests to evaluate the extent to which it would degrade. (see Figure 9, C). As can be observed, the sample has decomposed.

A repeat R6G sample, #2, (see Figure 10) was also irradiated, and similar scintillation and decomposition was observed.

Analysis of these samples was done to determine if the by-products impact any of the water quality requirements noted in the first report. Originally, the R6G sample had the following characteristics: pH: 6.00, conductivity: 4.5 µS/cm, turbidity: 0.92 NTU. After exposure, the sample was much more acidic, with greater conductivity but increased turbidity: pH: 4.61, conductivity: 17.1 µS/cm, turbidity: 2.11 NTU. This implies the decomposition would render the solution a problem for the stainless-steel coatings. Additional work to understand the impact of these by-products is underway.

Figure 9. Images of the R6G sample #1 during testing (A), after 5 min irradiation (B), and after 360 min irradiation (C).

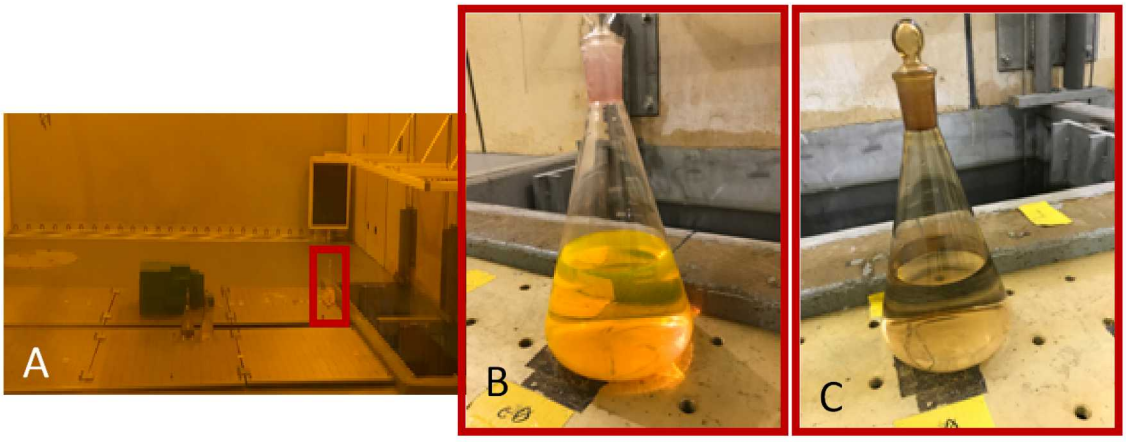


Figure 10. Images of R6G sample #2 during testing (A), before irradiation (B), and after 10 min irradiation (C).

(b) TAD. Again, a TAD solution was made at the concentration levels of the previous BRUCE testing and placed in the GIF irradiation chamber (Figure 11). Prior to irradiation, the sample was white and easily dispersed as shown in Figure 11, B. The sample was then exposed for 6 hours to the ^{60}Co sources, and result is shown in Figure 11, C. Several observations were made during the process. First, the glassware turned a dark color during this exposure, which is thought to be due to the changes in the metals co-located in the glass. However, the TAD solution did not show any color changes or additional precipitation during the exposure. Further, the solution's properties were only slightly altered. The pre-irradiation properties were: pH: 6.51, conductivity: 3.3 µS/cm, turbidity: 164 NTU. Post-irradiation properties were: pH: 6.00, conductivity: 4.3 µS/cm, turbidity: 181 NTU. The variations are all believed to be within experimental measurement error and fall within the parameters set out in the original report. In fact, the turbidity increased, which would be a useful property for the obscurant objective.

Figure 11. Images of the TAD sample (A) during testing, (B) before irradiation, and (C) after 360 min irradiation.

(c) CBOD. A sample of BRUCE-testing-equivalent CBOD solution was prepared and placed in the GIF by the source pool (Figure 12, A). The initial properties of the CBOD solution (Figure 12, B) were: pH: 5.80, conductivity: 76.5 µS/cm, turbidity: 9.17 NTU. After a 10-minute exposure, the sample was clear with a discolored flask. The solution had altered properties at: pH: 5.30, conductivity: 31.6 µS/cm, turbidity: 7.38 NTU. Not unexpectantly, the organic dye rapidly decomposed. Additional properties of the final organic species generated are being evaluated.

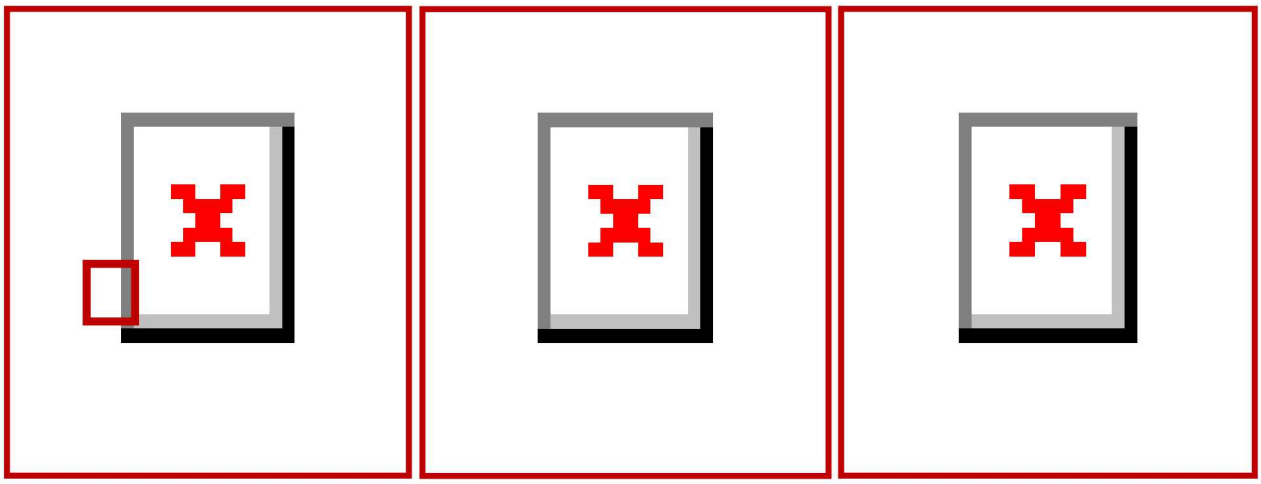


Figure 12. Images of the CBOD sample (A) during testing, (B) before irradiation, and (C) after 10 min irradiation.

(d) DEA. A sample of DEA at the concentration used in BRUCE testing was exposed at the same time as the CBOD. Figure 13 shows the sample placement (A), the sample before irradiation (B), and post irradiation (C). The solution properties prior to irradiation were: pH: 5.20, conductivity: 19.6 µS/cm, turbidity: 86.0 NTU. Post-irradiation properties were: pH: 5.24, conductivity: 9.9 µS/cm, turbidity: 92.3 NTU. Results show minimal effects from this radiation exposure, and all parameters are still within acceptable limits.

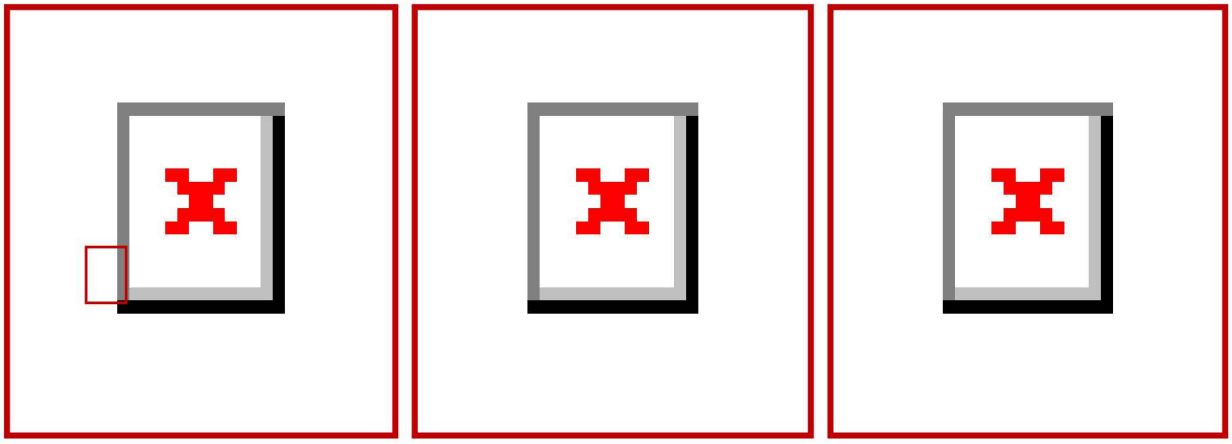


Figure 13. Images of the DEA sample (A) during testing, (B) before irradiation, and (C) after 10 min irradiation.

4. SUMMARY AND CONCLUSION

Potential commercially-available obscurants for use in ^{60}Co storage pools in IRFs were further evaluated for corrosive behavior on C-188 pencil stubs and retention of obscurant properties under high-radiation exposure (^{60}Co at ~207 rad/s). The results from these studies reveal: 1) the obscurants had no impact on the stainless-steel pencil slugs and some may even assist in terms of corrosion resistance and 2) organic obscurant species readily decompose when irradiated, whereas the ceramic materials survived intact.

The potential obscurants studied included: titania aqueous dispersions (TAD – water soluble white paint), Chlorazol Black (CBOD – Chlorazol Black organic dye), powdered milk (COW – calcium obscurant in water), diatomaceous earth (DEA – diatomaceous earth additive), and rhodamine 6G (R6G). For corrosion efforts, stubs from an inert C-188 source rod were soaked in obscurant solutions and visually inspected. For radiation stability, obscurant samples were exposed to ^{60}Co radiation sources.

REFERENCES

[1] Boyle, T. J., P. C. Reuel, W. D. Reinholtz, M. P. Romero, A. D. Thompson, L. J. Gilbert, M. Kuca, and A. W. Cook. 2019. "SAND2019-7727: Evaluation of Commercially Available Precursors for Wet Industrial Irradiator Facility Pools to Delay Adversarial Source Removal."

[2] Chmielewski, A. G. 2006. Gamma Irradiators for Radiation Processing (Report number INIS-XA--862. Austria: International Atomic Energy Agency, 1-46.

APPENDIX A. CORROSION EXPOSURE DATA

Each beaker was filled with 300 mL of deionized (DI) water and each obscurant at a concentration as that of the large-scale BRUCE tests. "Before" pictures were taken of the stainless-steel slugs, as well as, at intervals of 5 minutes, 30 minutes, 1, 21, 46, 147 and 188 hours. BRUCE beaker initial testing was done without magnified optical imaging. Initial images are shown in Figure A-1, with 'aged' images shown in figures A-2 through A-8.

Figure A-1. Before image of slugs and solutions observed.

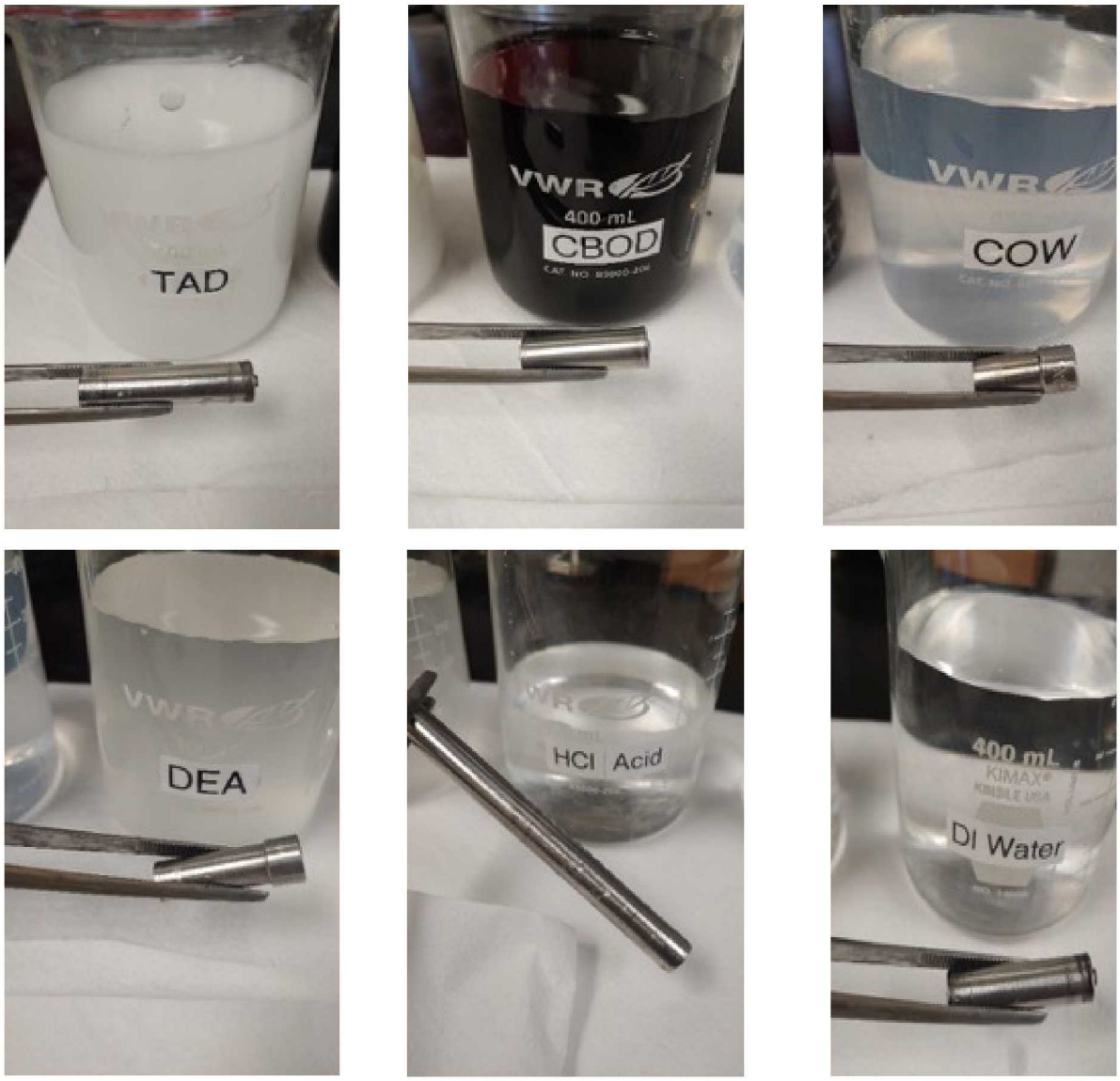


Figure A-2. Observation of slugs at the 5 min interval.

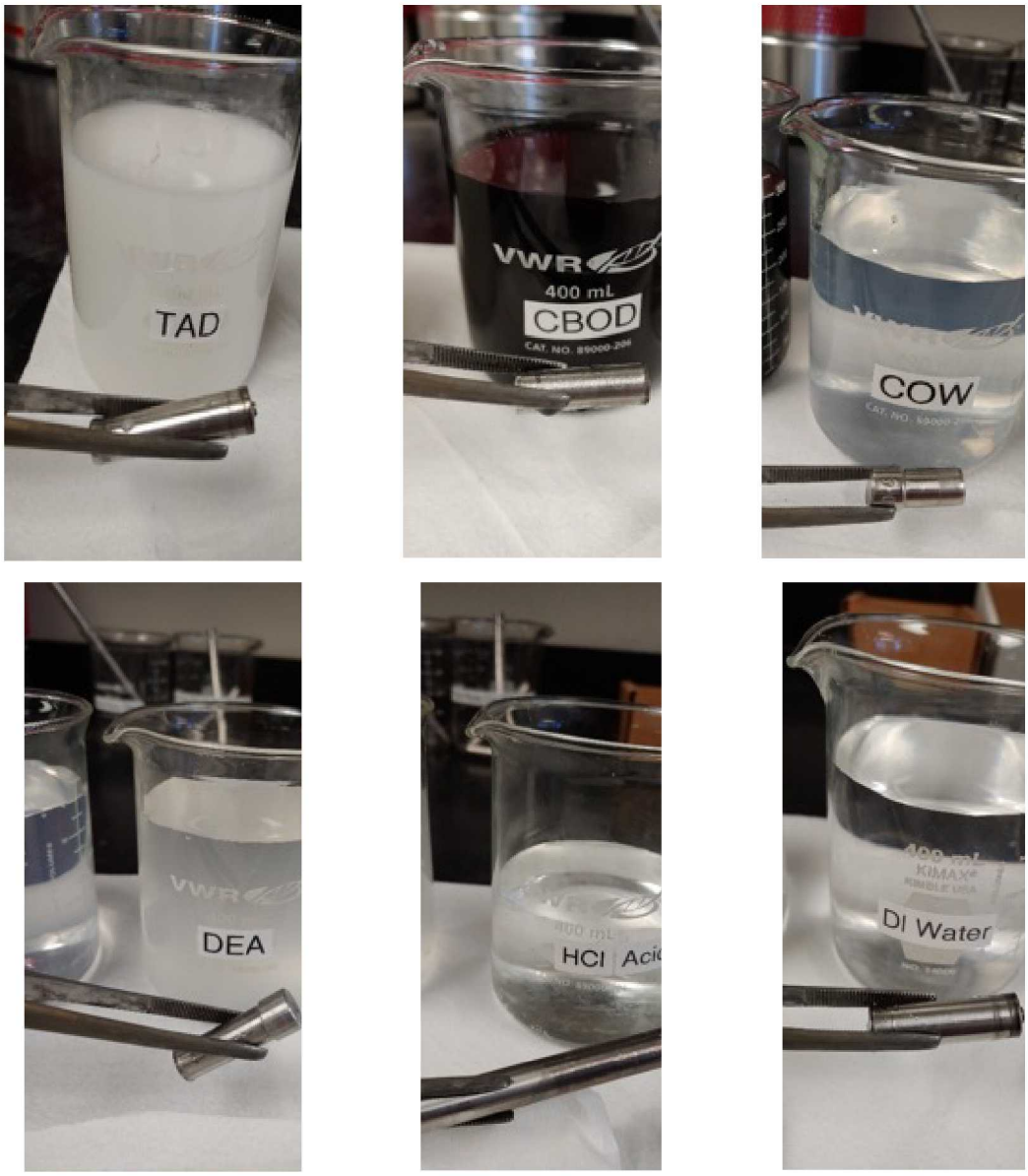


Figure A-3. Observation of slugs at the 30 min interval.

Figure A-4. Observation of slugs at the 1 h interval.

Figure A-5. Observation of slugs at the 21 h interval.

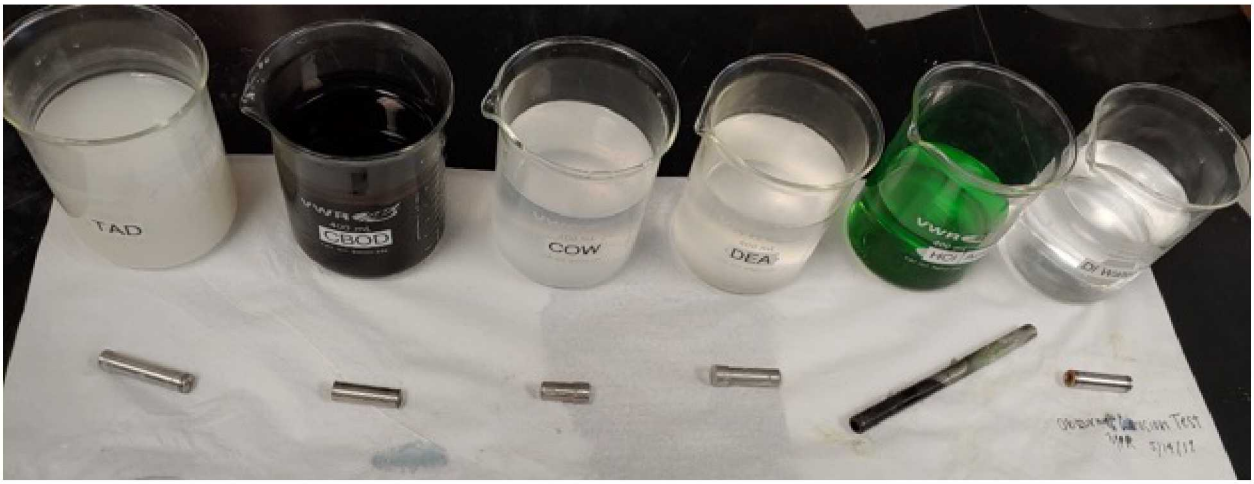


Figure A-6. Observation of slugs at the 46 h interval.

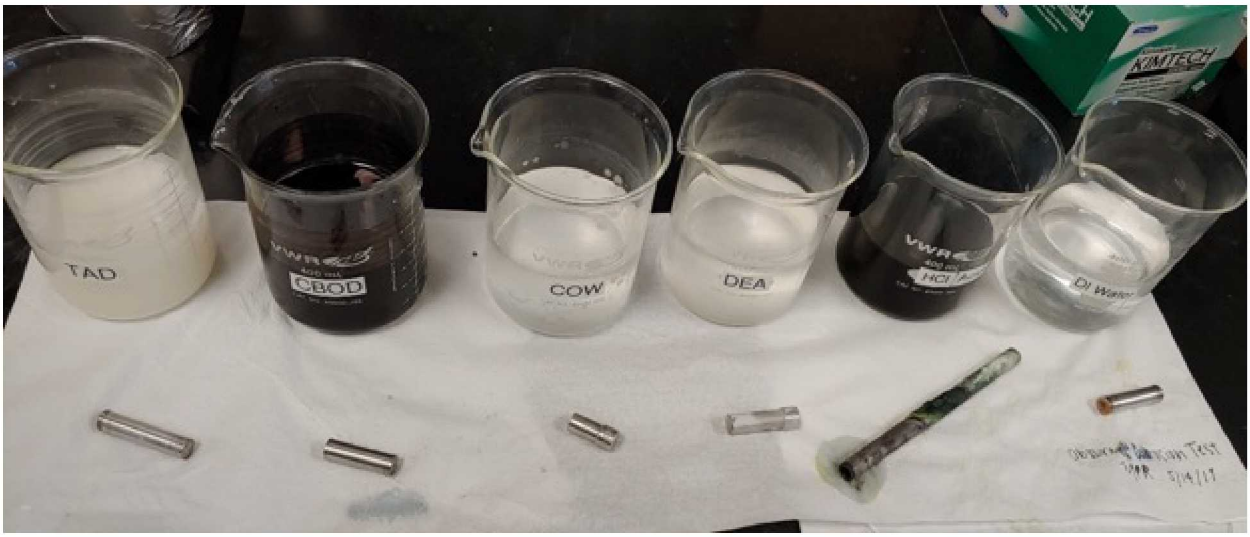


Figure A-7. Observation of slugs at the 147 h interval.

Figure A-8. Observation of slugs at the 188 h interval.

A small-scale reaction to further observe changes wrought by the BRUCE obscurants was undertaken. Three stainless-steel slugs were immersed separately in the three samples: TAD, DI water and concentrated HCl acid. These samples were chosen due to the way they reacted or did not react in the previous data.

To monitor any induced corrosion a microscope was used to take optical images at 100 times magnification. The microscope used was a Keyence VHZ 7000 optical microscope. Pictures were taken at various areas along the slugs. Figure A-9 shows the "before" pictures and the solutions. The areas photographed on the slugs each time are depicted in Figure A-10 by a red dot.

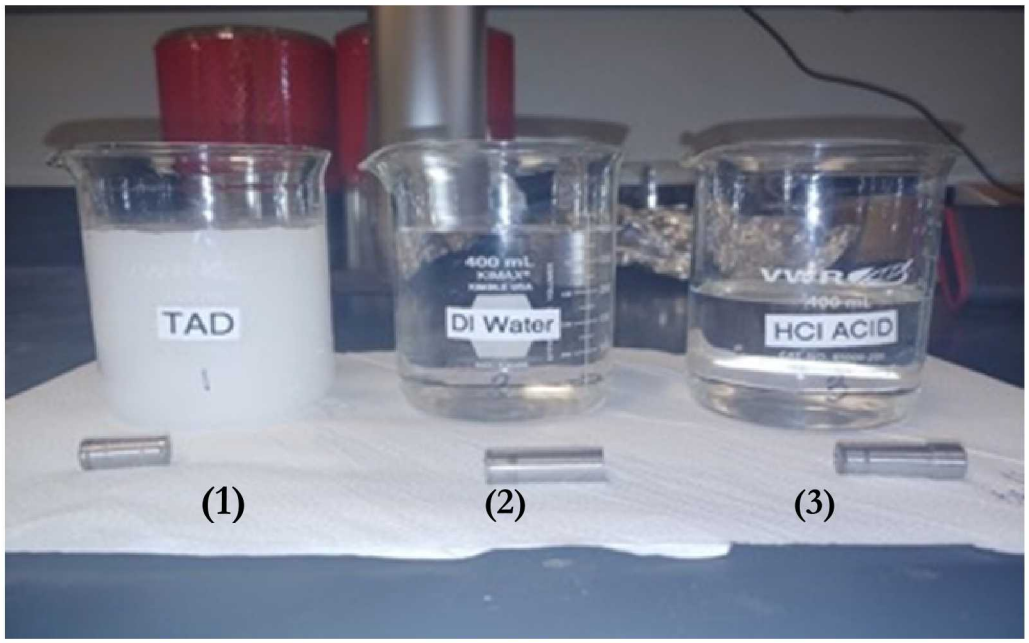


Figure A-9. Before picture of stainless-steel slugs and samples: (1) TAD, (2) DI water, and (3) concentrated HCl. Numbered to refer to stainless-steel slugs depicted in Figure A-10

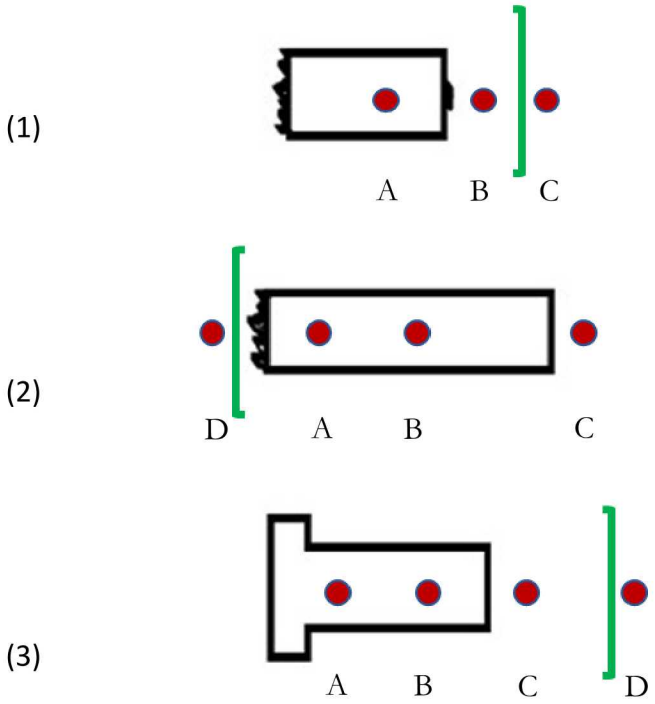


Figure A-10. Schematic of slugs (1 – 3) and locations (●) of where pictures were obtained. Green bar indicates face (90°) picture taken.

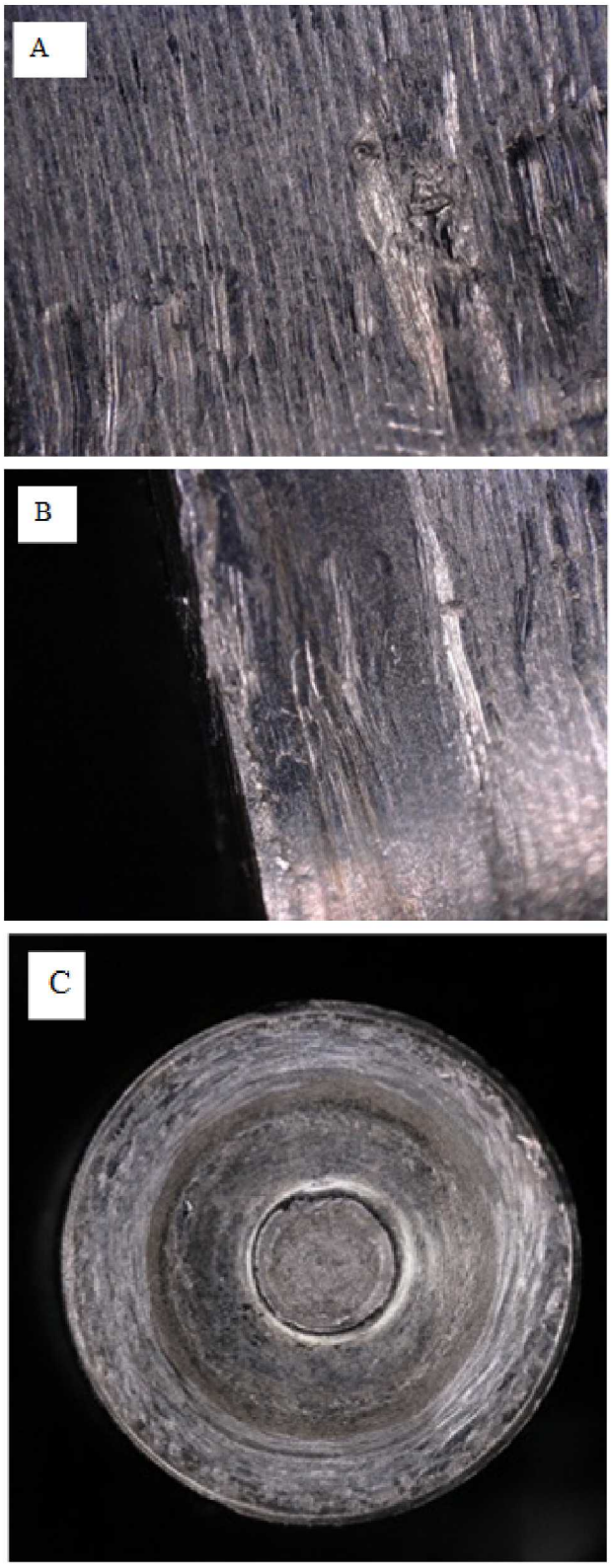


Figure A-11. Optical image of slug 1 at spots: A, B, and C (time 0)

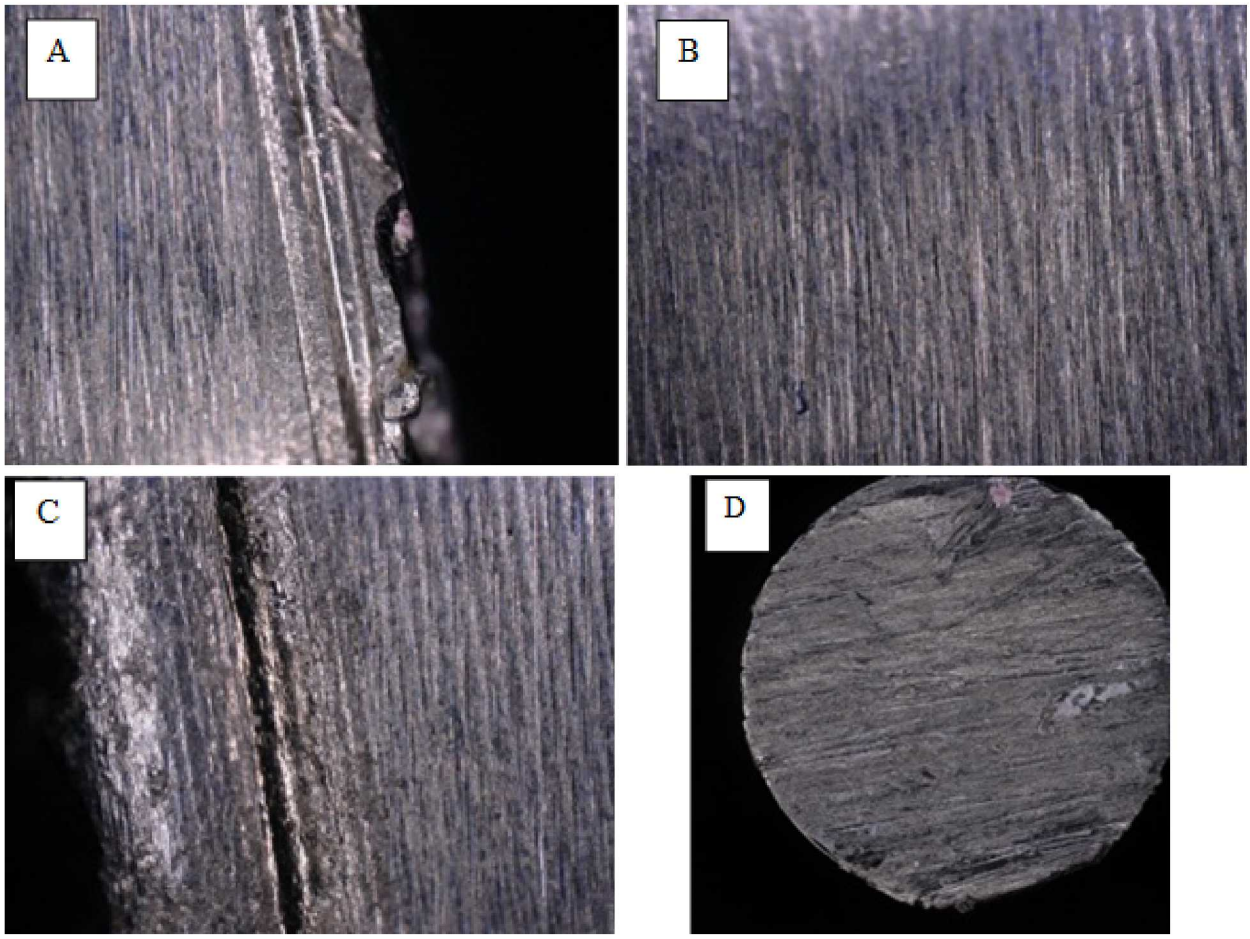


Figure A-12. Optical image of slug 2 at spots: A, B, C and D (time 0).

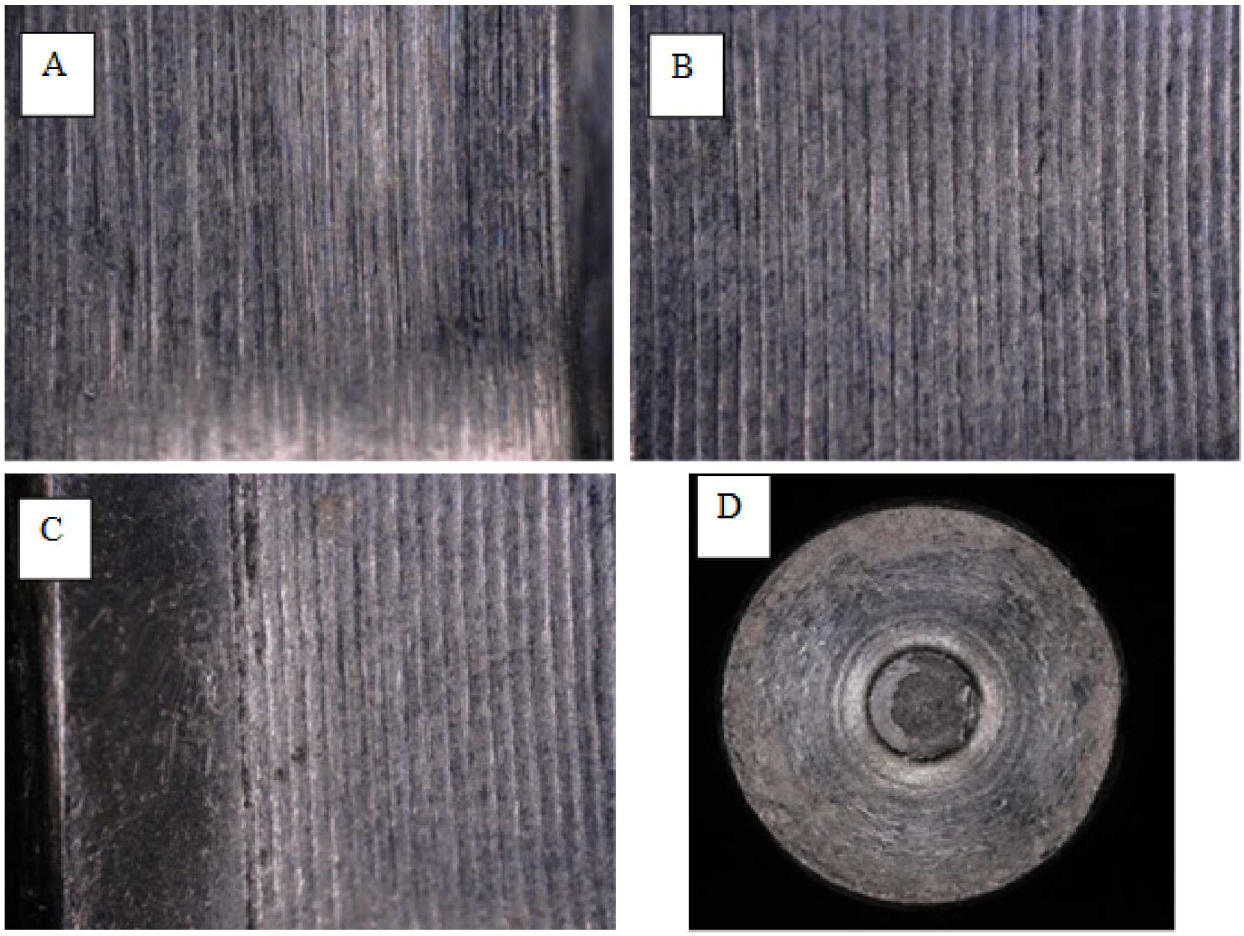


Figure A-13. Optical image of slug 3 at spots: A, B, C and D (time 0).

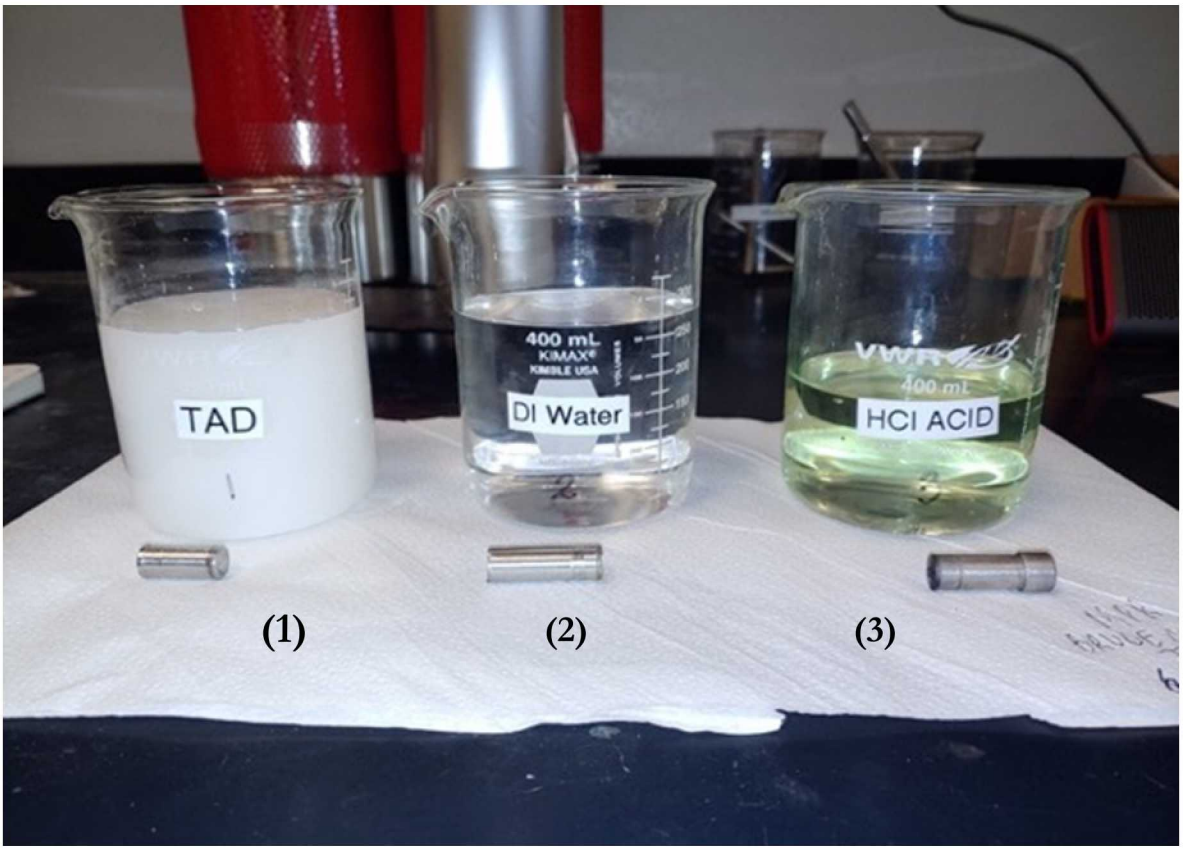


Figure A-14. Slugs at 46 h: (1) TAD, (2) DI water, and (3) concentrated HCl acid.

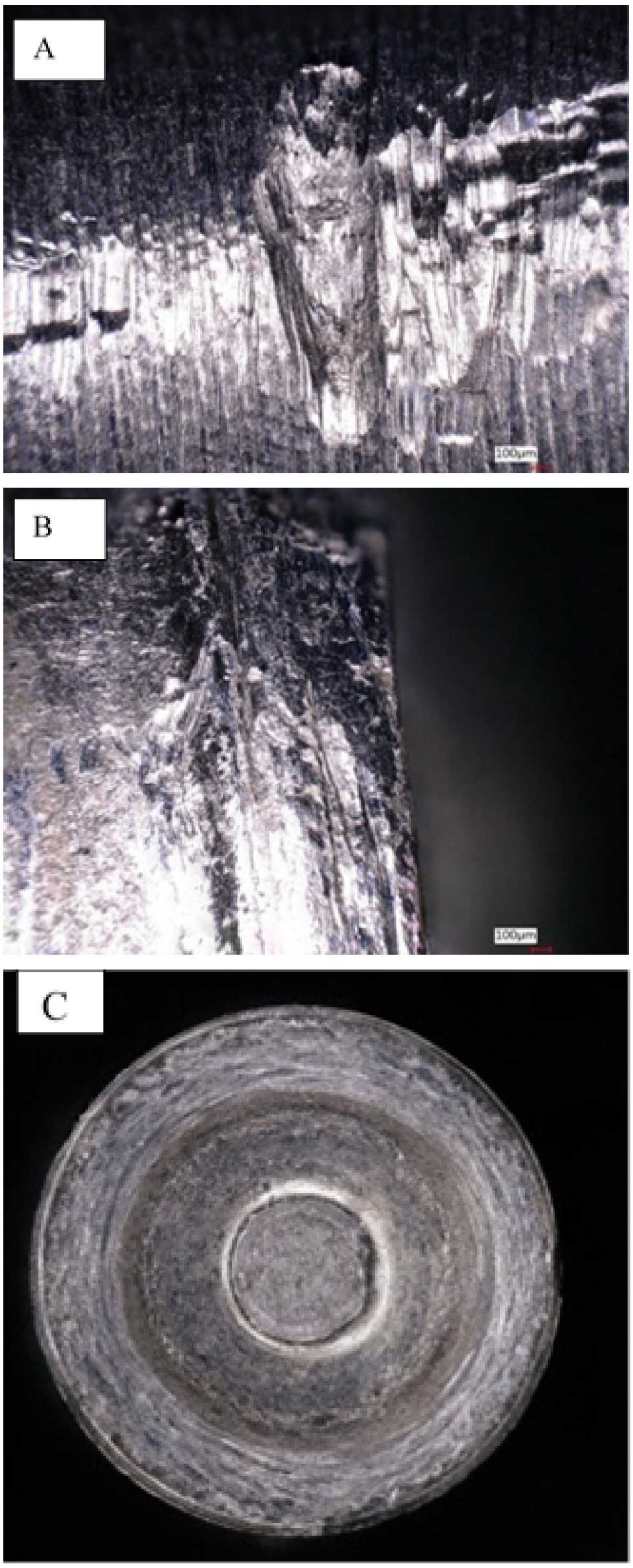


Figure A-15. Optical image of slug 1 in TAD at spots: A, B, and C (time 46).

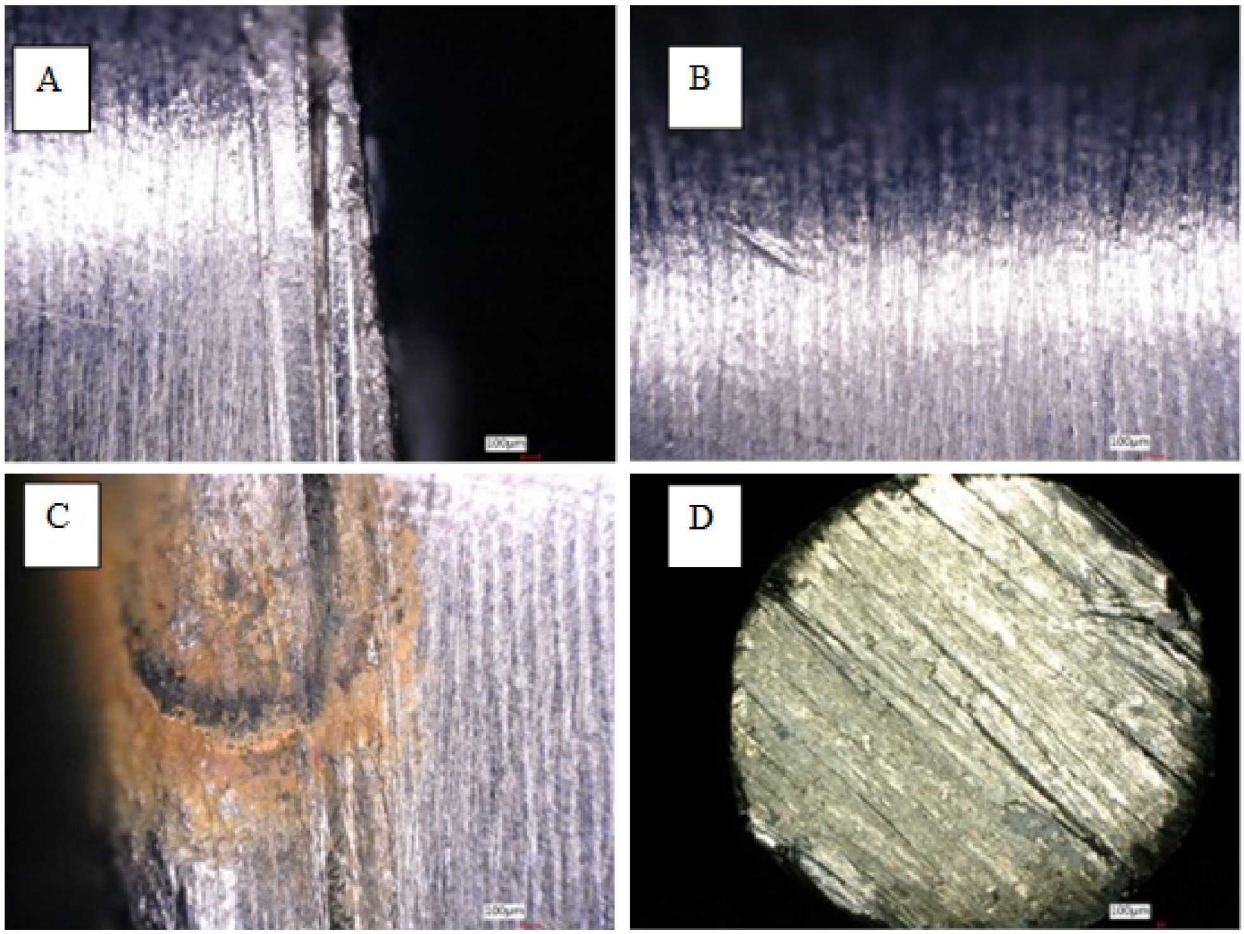


Figure A-16. Optical image of slug 2 in H_2O at spots: A, B, C and D (time 46h).

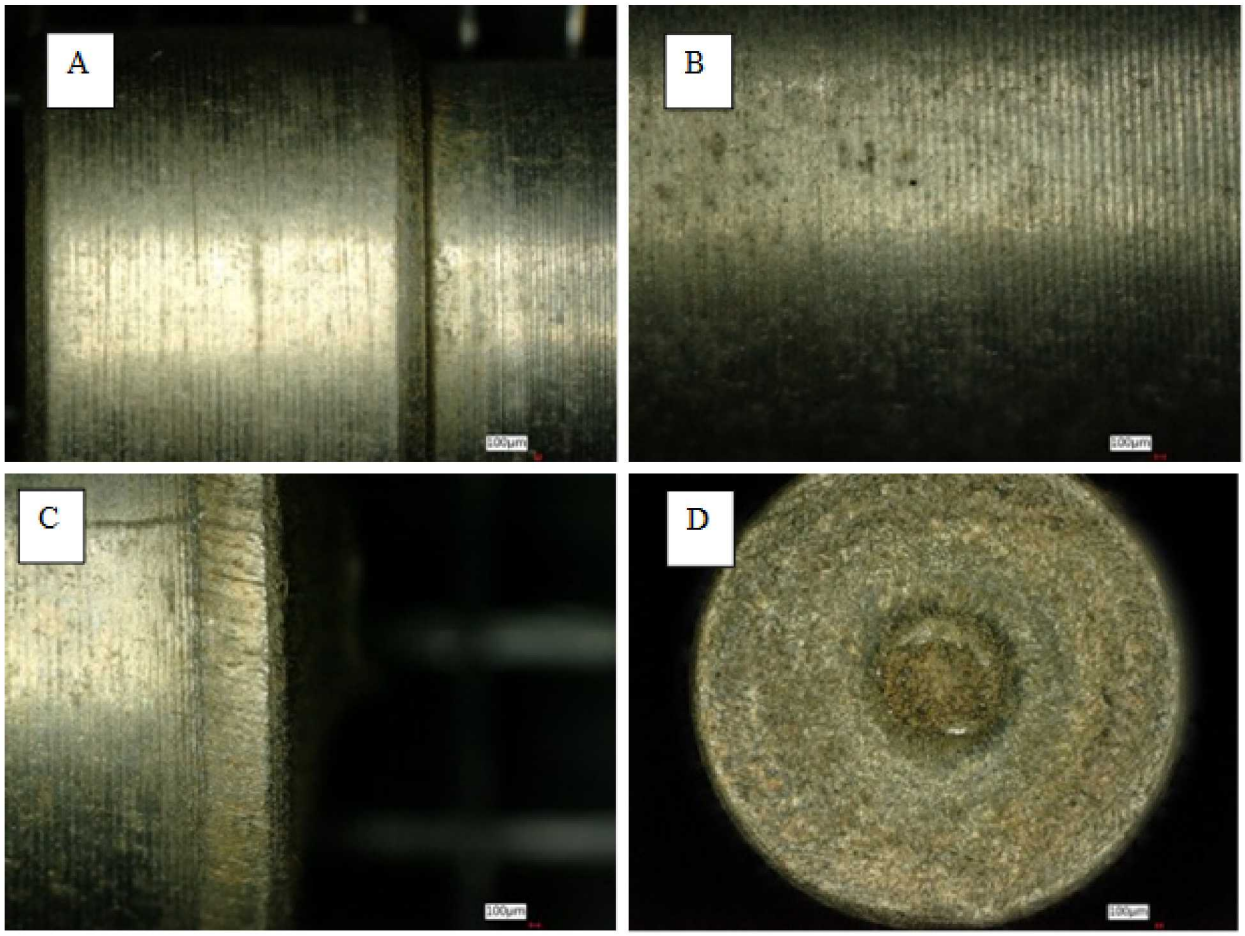


Figure A-17. Optical image of slug 3 in HCl at spots: A, B, C and D (time 46h).

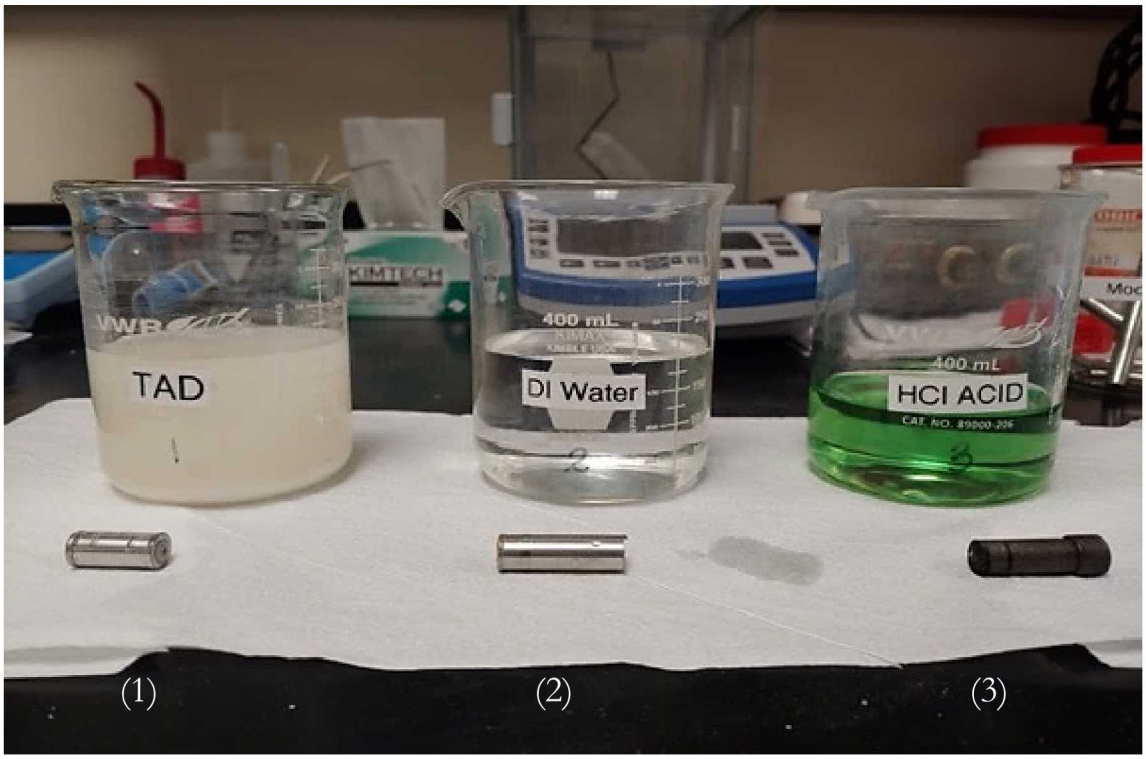


Figure A-18. Slugs at 178 h: (1) TAD, (2) DI water, and (3) concentrated HCl acid.

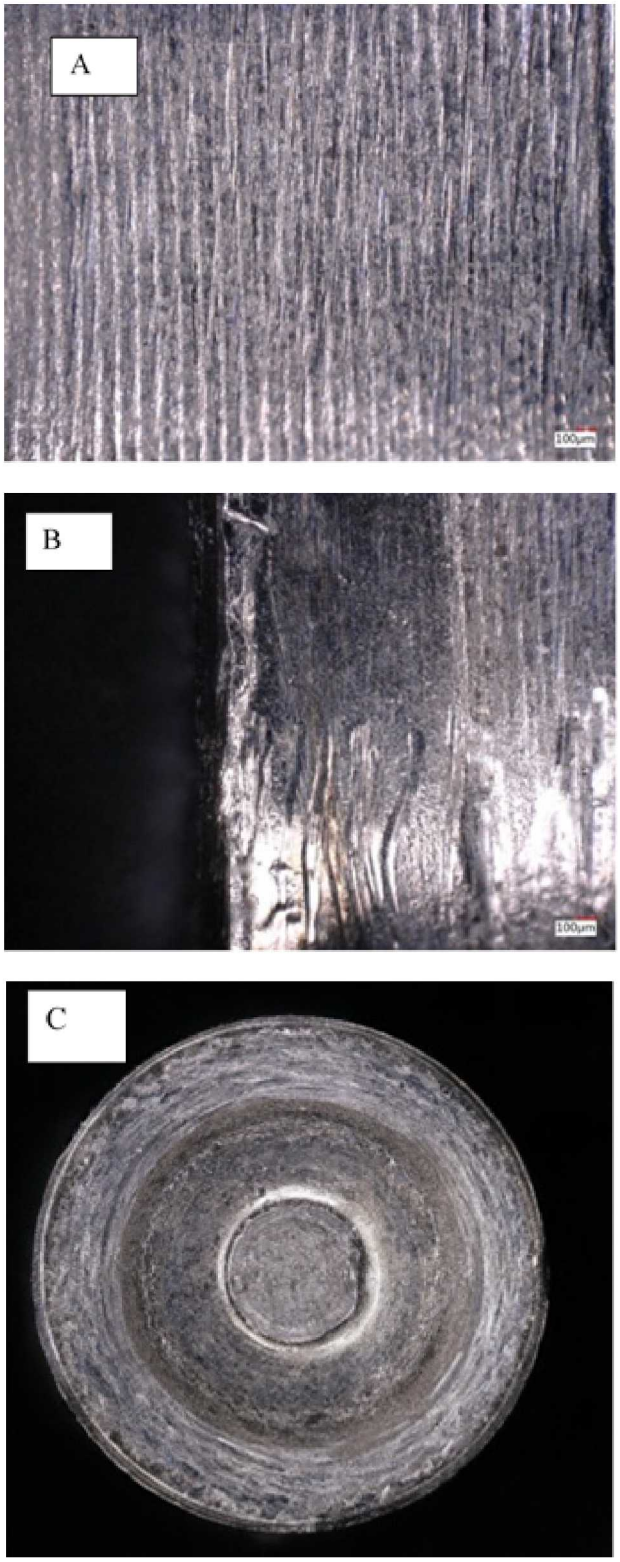


Figure A-19. Optical image of slug 1 in TAD at spots: A, B, and C (time 178 h).

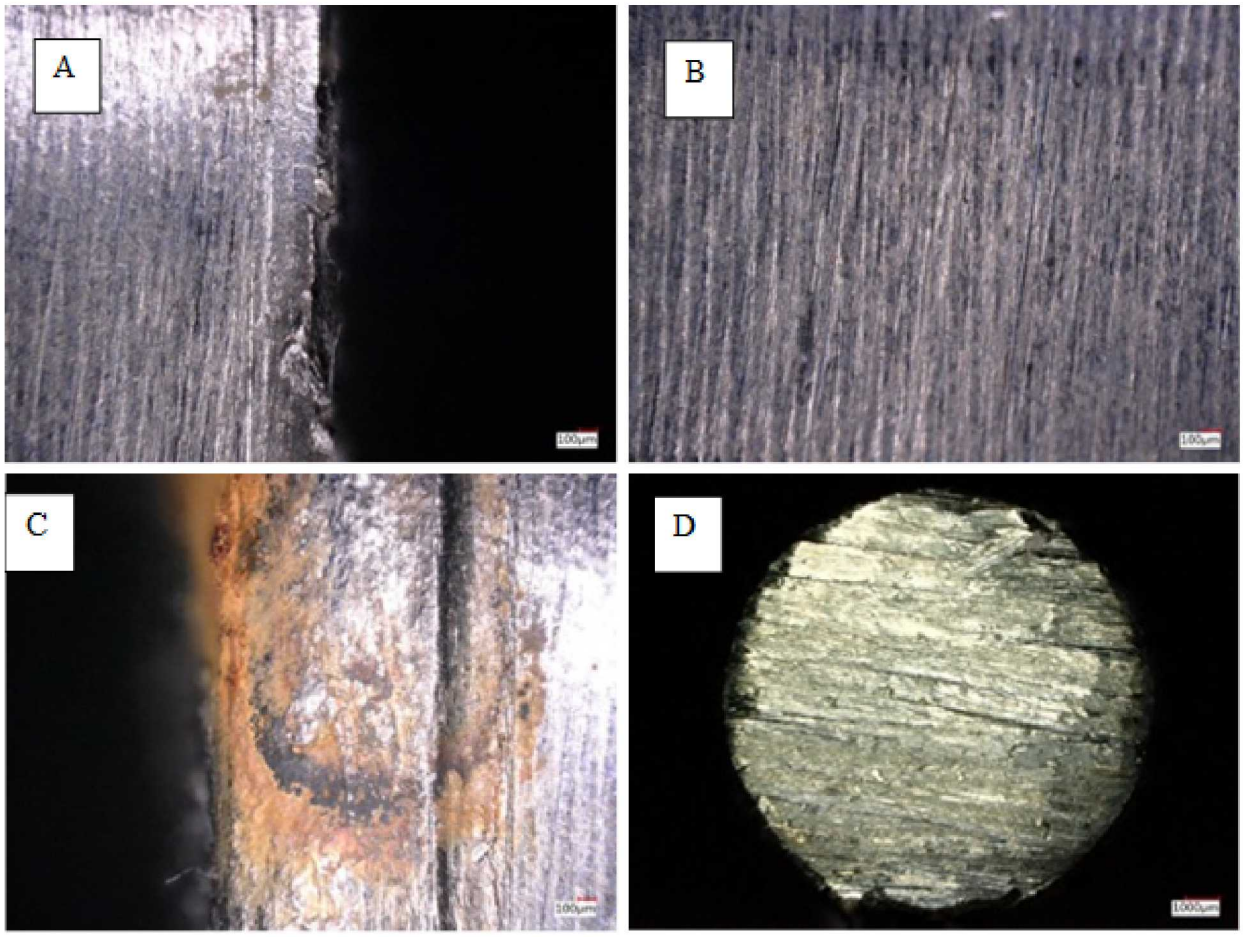


Figure A-20. Optical image of slug 2 in H_2O at spots: A, B, and C (time 178 h).

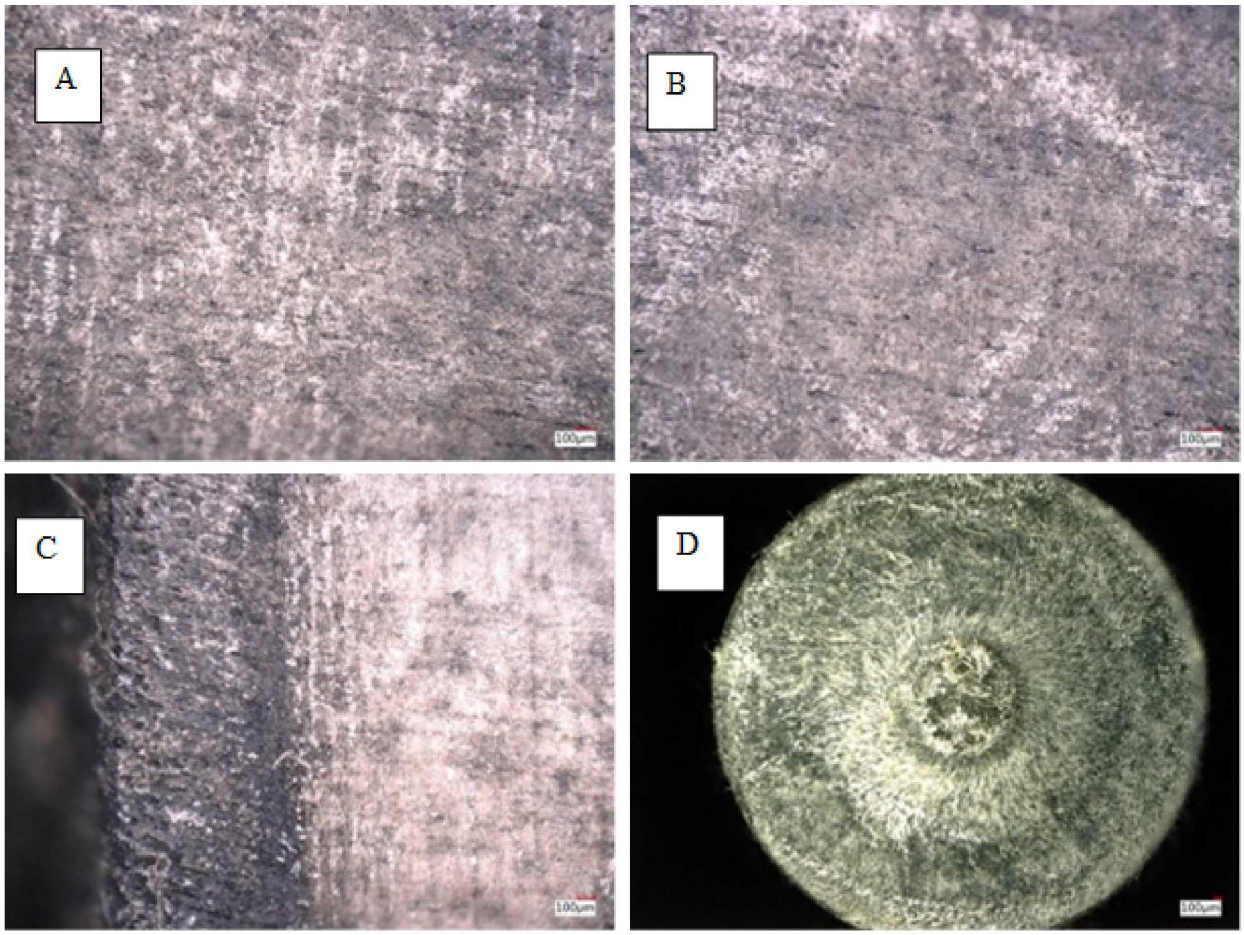


Figure A-21. Optical image of slug 3 in HCl at spots: A, B, C and D (time 178 h).

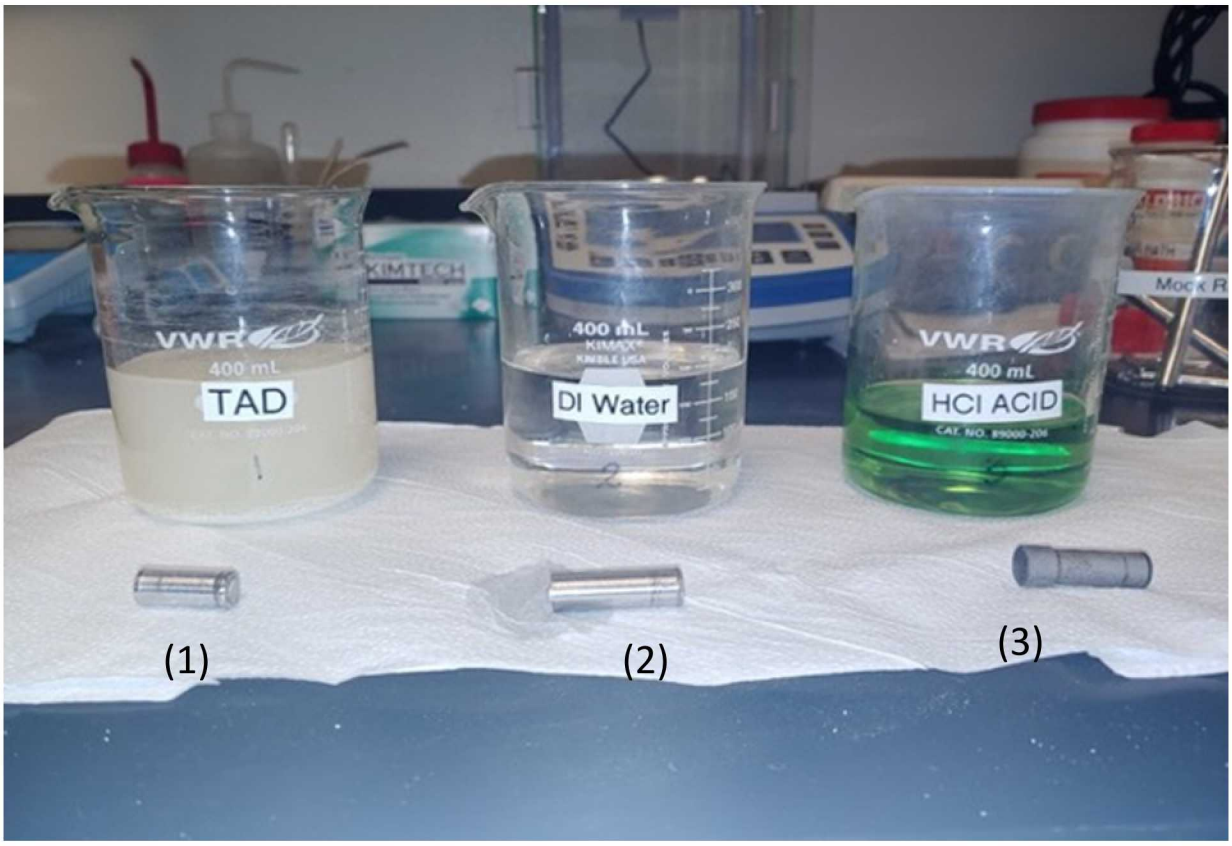


Figure A-22. Slugs at 198 h: (1) TAD, (2) DI water, and (3) concentrated HCl acid.

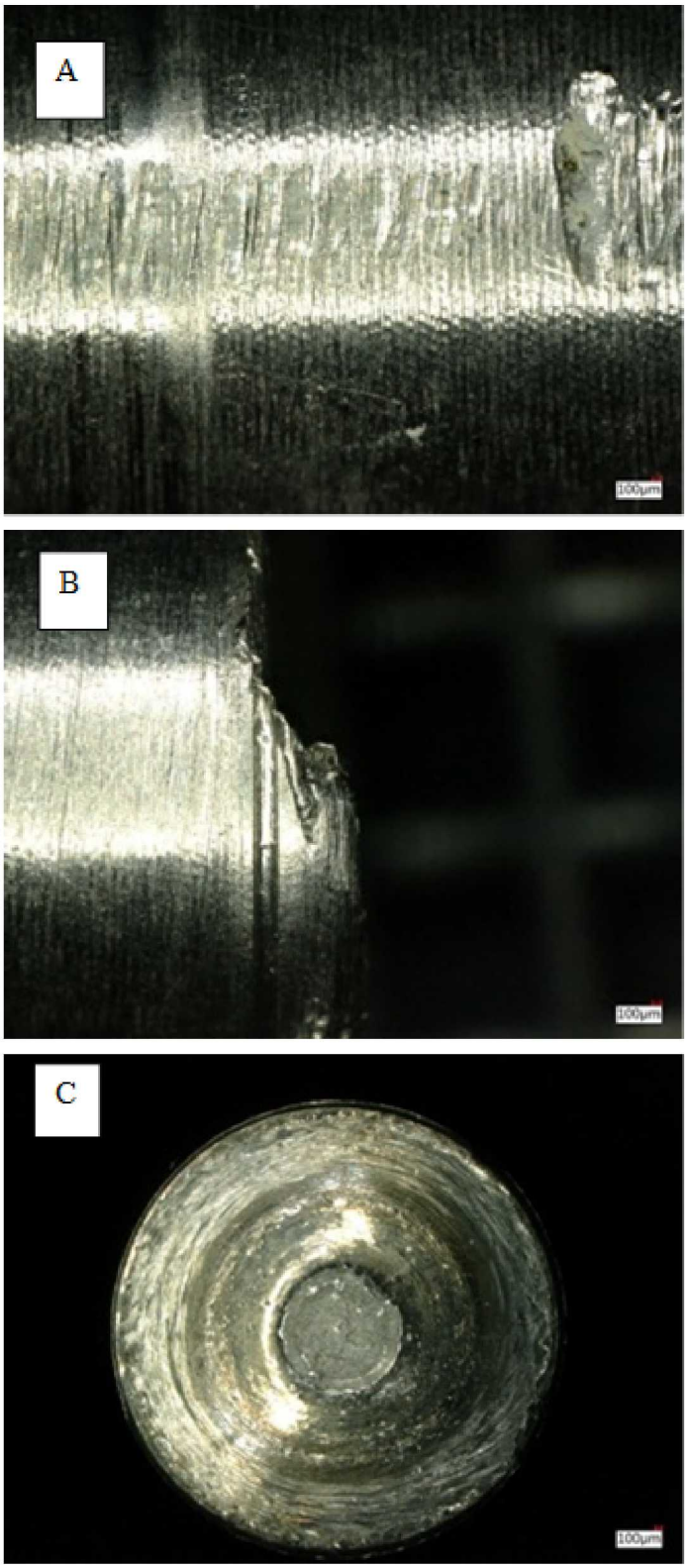


Figure A-23. Optical image of slug 1 in TAD at spots: A, B, and C (time 196 h).

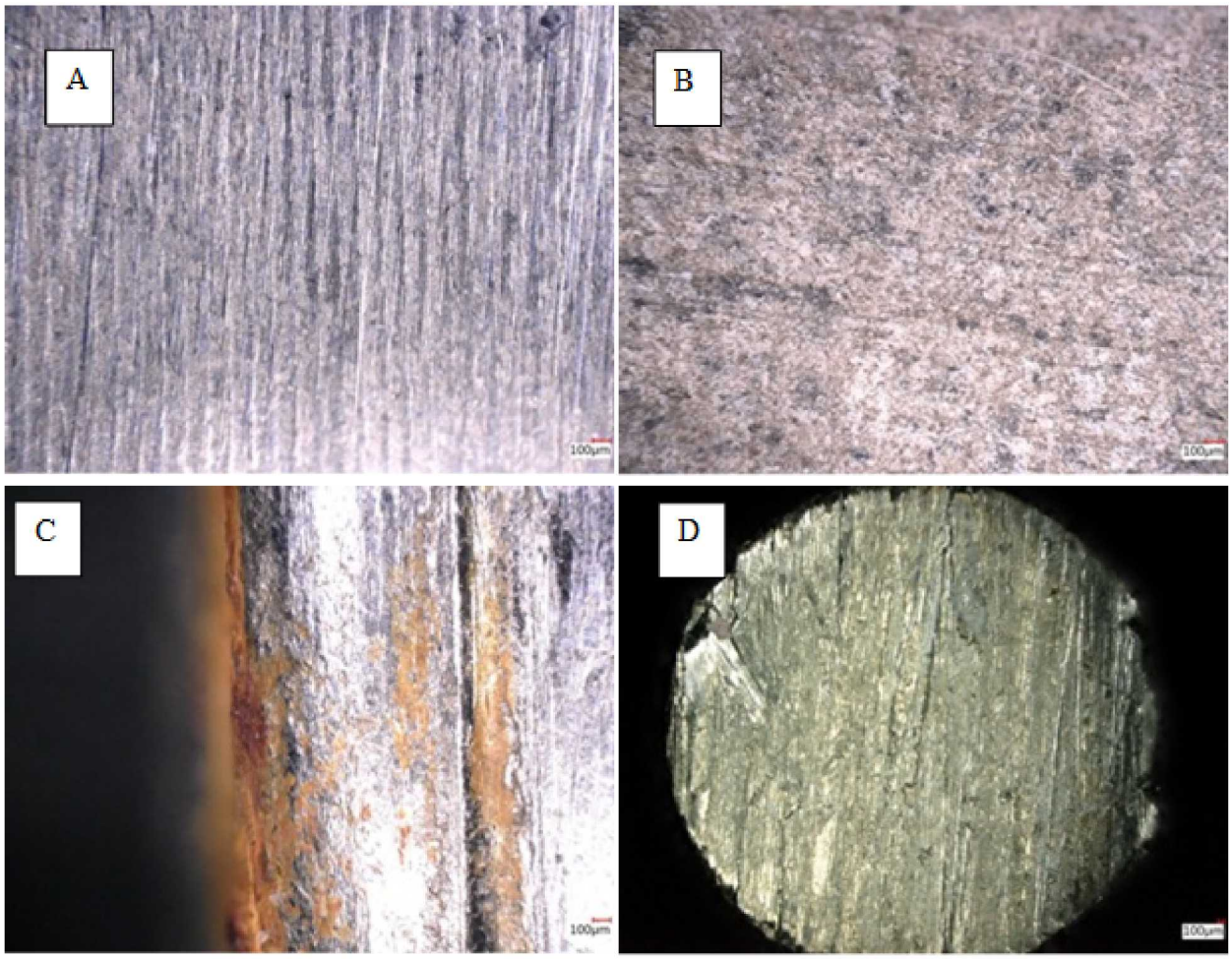


Figure A-24. Optical image of slug 2 in H_2O at spots: A, B, C, and D (time 196 h).

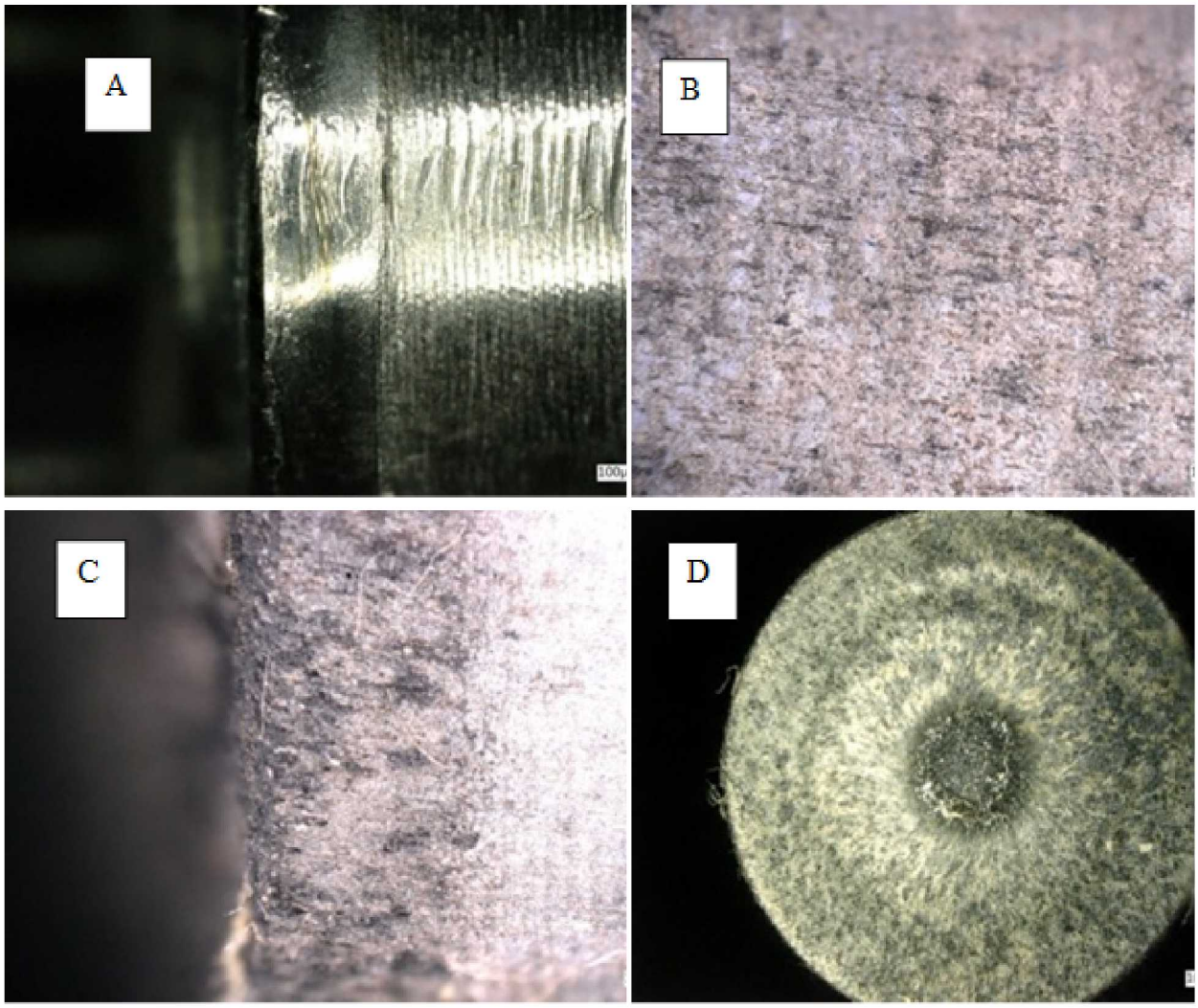


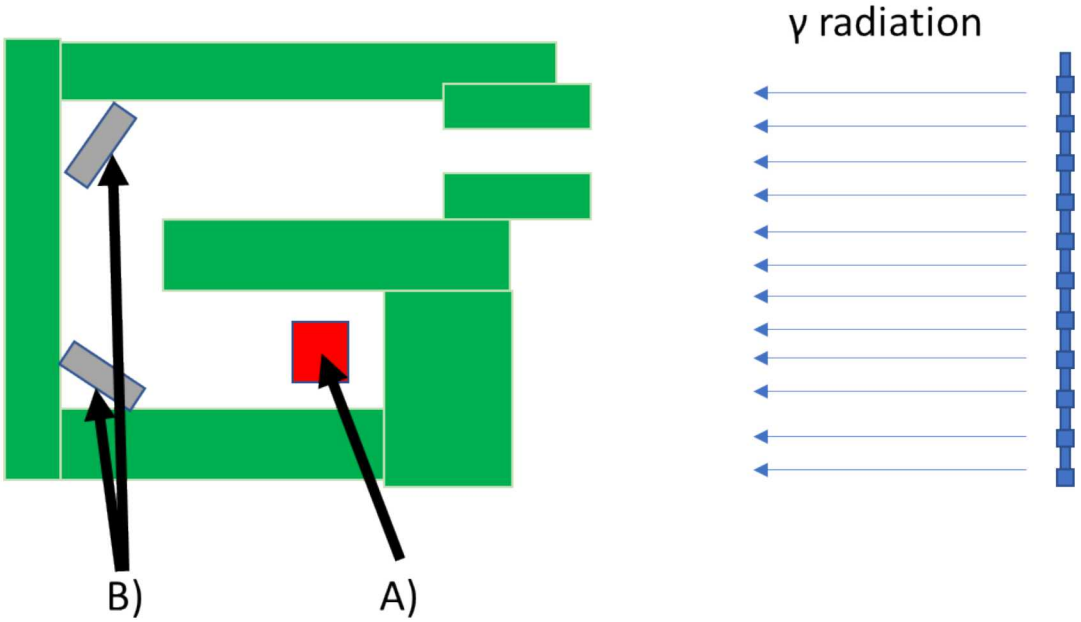
Figure A-25. Optical image slug 3 in HCl at spots: A, B, C and D (time 196 h).

APPENDIX B. RADIATION EXPOSURE DATA

Table B - 1. Turbidity, pH, and Conductivity Before and After Irradiating Samples

Obscurant	Turbidity (NTU)		pH		Conductivity (μS/cm)	
	Pre	Post	Pre	Post	Pre	Post
R6G	0.92	2.11	6.00	4.61	4.5	17.1
TAD	164	181	6.51	6.00	3.3	4.3
CBOD	9.17	7.38	5.80	5.30	76.5	31.6
DEA	86.0	92.3	5.20	5.24	19.6	9.9

(a)



(b)

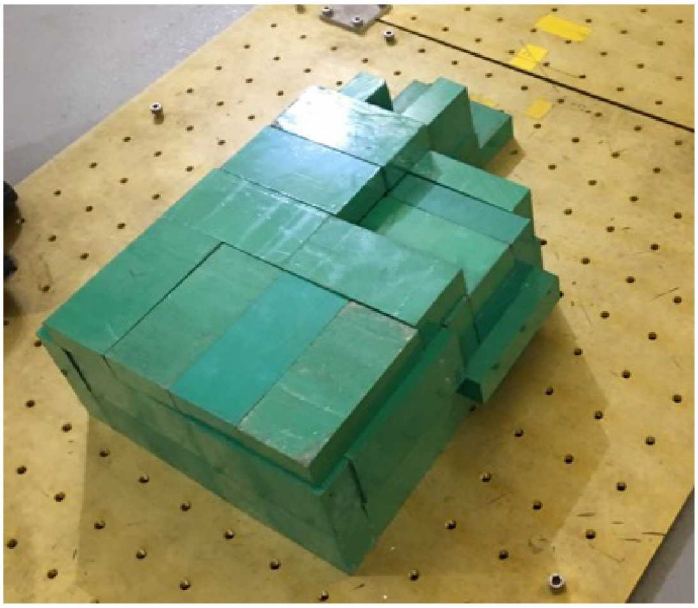
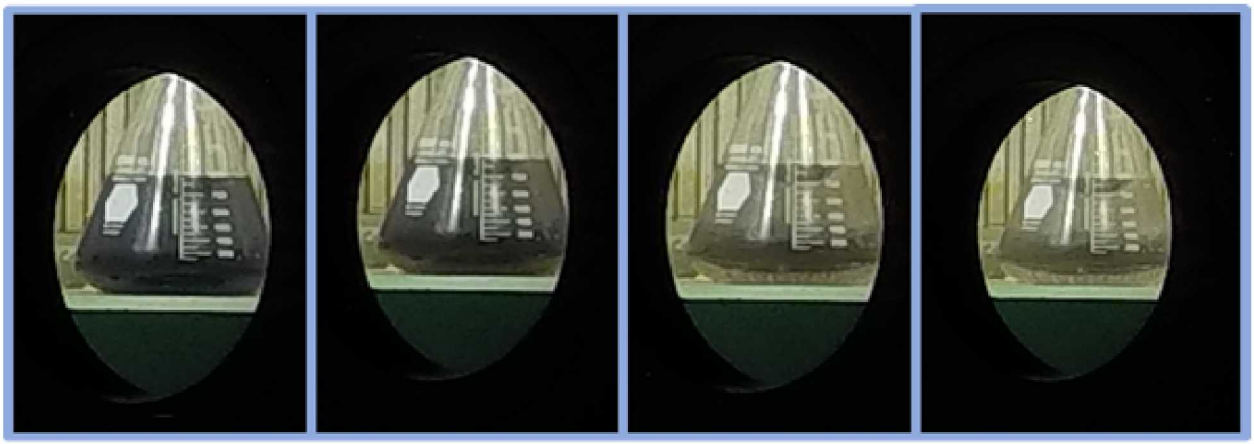
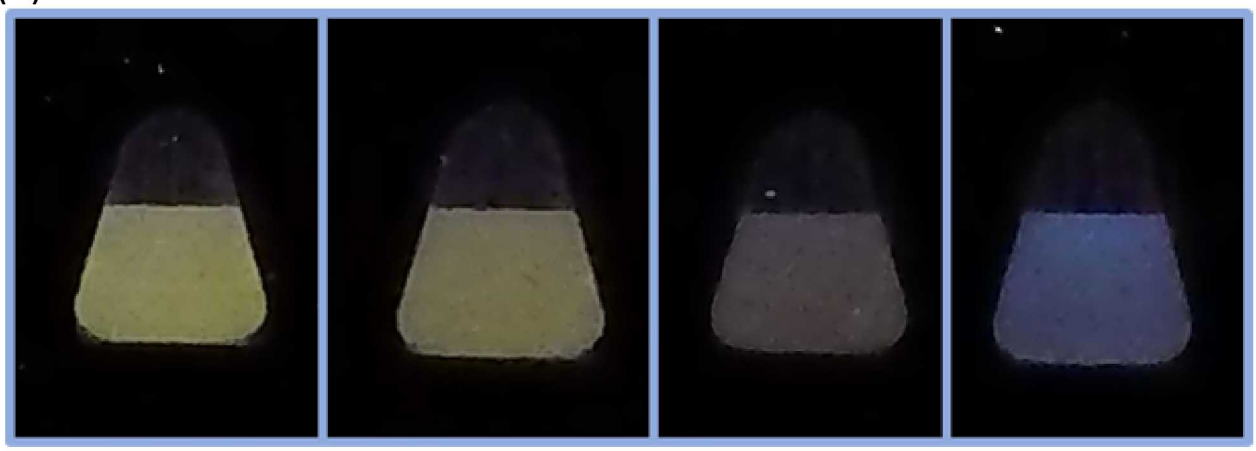


Figure B - 1. Design for protecting camera from source unitizing lead bricks (green): (a) schematic where A) GoPro™ and B) optical metallic mirror; and (b) testing apparatus.

(a)



(b)



(c)

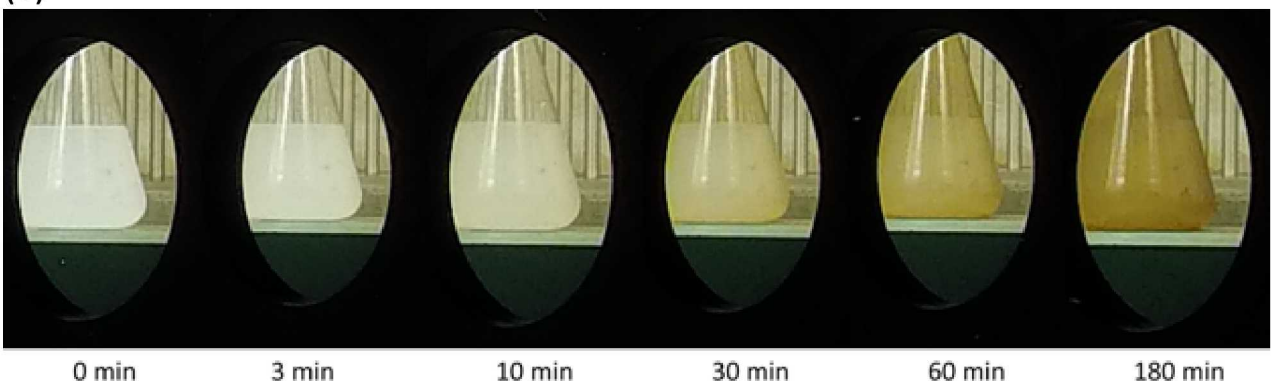


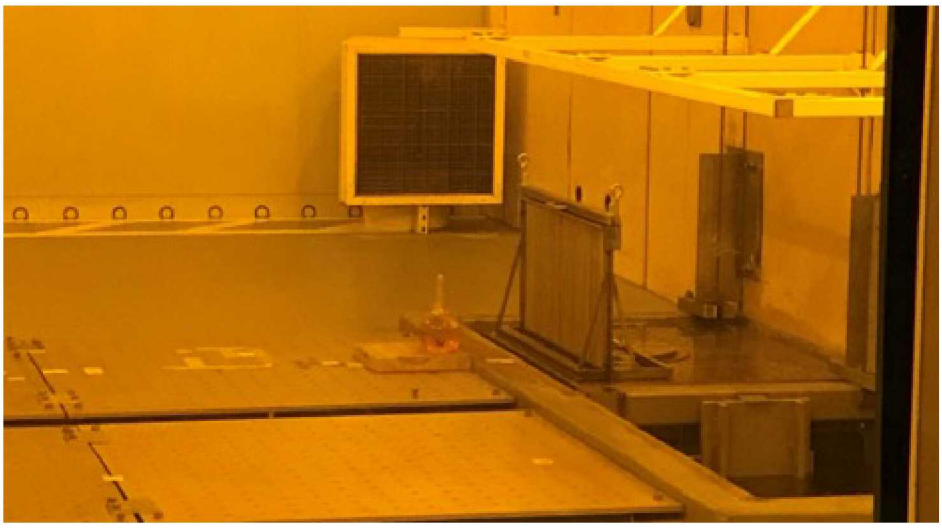
Figure B - 2. Optical Decay of Obscurants: (a) CBOD, (b) R6G, and (c) TAD

(a)

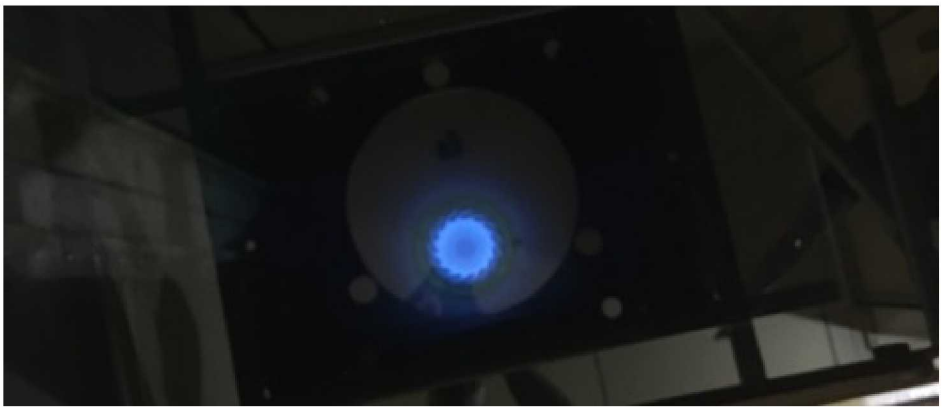
(b)

Figure B - 3. (a) Before and (b) after comparison of obscurants visually

(a)



(b)



(c)

Figure B - 4. Candid pictures of GIF testing: (a) sample testing exposure, (b) fuel in pool, (c) assembling the camera protection

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