Bosch Carbon Catalyst Regeneration in Water III: Experiments for and Related to Thermal Shock

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Bosch technology has the potential to close the atmospheric revitalization loop by providing theoretically 100% regeneration of metabolic carbon dioxide into oxygen. The main obstacle to using Bosch technology is over-pressurization issues in the carbon formation reactor. Prior work on Bosch catalyst regeneration established that agitating carbon coated beads in water broke up pressure causing clogs and regenerated the catalyst surface. This process proved to be time consuming with experiments taking at least one hour to regenerate ten grams of carbon coated beads. In an attempt to shorten the processing time, thermally shocking the carbon coated beads prior to mechanical agitation was tested. Agitating the carbon coated beads after exposure to a saturated steam environment was also tested to ensure that the steam generated by thermally shocking the carbon coated beads would not cause adverse effects. The rationale behind thermally shocking the carbon coated beads is that the bead would pull away from the carbon shell due to a difference in the thermal expansion coefficients. The results of this project show that the processing time can be cut in half when compared to previous work, and that saturated steam has little to no effect on regeneration.

Acronyms and Nomenclature

AR	=	Air Revitalization
CFR	=	Carbon Formation Reactor
CHX	=	Condensing Heat Exchanger
COR-CaTS	=	Carbon Dioxide Reduction Catalyst Test Stand
DI	=	Deionized
ECLSS	=	Environmental Controls and Life Support Systems
ISRU	=	In-Situ Resource Utilization
ISS	=	International Space Station
LEO	=	Lower Earth Orbit
OGA	=	Oxygen Generation Assembly
PIG	=	Pipeline Inspection Gage
RWGS	=	Reverse Water Gas Shift
RWGSR	=	Reverse Water Gas Shift Reactor
S-Bosch	=	Series Bosch
SoA	=	State of the Art
WPA	=	Water Processing Assembly
α	=	average mass reduction

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I. Introduction

THE Atmospheric Revitalization (AR) system handles multiple functions including trace contaminant control, carbon dioxide removal, and oxygen regeneration. The oxygen regeneration system is used to reclaim oxygen from metabolic carbon dioxide. The current State of the Art (SoA) technology for oxygen regeneration is the Sabatier reactor which has been tested on the International Space Station (ISS). The Sabatier reactor converts carbon dioxide and hydrogen into water and methane by the reaction detailed in Equation 1. The methane is vented overboard, and the water is sent to the Water Processing Assembly (WPA) which is cycled through the closed loop water system until it returns to the AR system through the Oxygen Generation Assembly (OGA) [1]. The OGA then splits the water into oxygen, which is sent to the cabin, and hydrogen, which is sent to the Sabatier reactor. Methane venting leads to a loss of hydrogen, an essential element of water and the Sabatier reaction (Equation 1), thus creating a need for water resupply. This need for water resupply constrains long duration space flight to Low Earth Orbit (LEO). To close the AR loop, technologies for the functions of methane post processing and the purification of the post processing effluent are currently being investigated [2], [3], [4].

Sabatier Reaction $$CO_2 + 4H_2 \rightleftarrows CH_4 + 2H_2O \quad (1)$$

An alternative solution for closing the AR loop would be to replace the Sabatier reactor with a Bosch process based system. The Bosch process employs the reactions listed in Equations 2-4 to create a system that could theoretically reclaim 100% of the oxygen in metabolic carbon dioxide with the products of elemental carbon and water. Technologies are currently being developed in the In-Situ Resource Utilization (ISRU) group to utilize this resource [5], [6], [7]. The Bosch process has yet to move beyond the development stage as a system due to historical issues with reactor efficiency, catalyst degradation, and over-pressurization issues [5], [8], [9], [10], [11].

Reverse Water Gas Shift (RWGS) Reaction $$CO_2 + H_2 \rightleftarrows CO + H_2O \quad (2)$$

CO Hydrogenation Reaction $$CO + H_2 \rightleftarrows C + H_2O \quad (3)$$

Boudouard Reaction $$2CO \rightleftarrows C + CO_2 \quad (4)$$

Efforts to solve the reactor efficiency issue resulted in a study that showed the Reverse Water Gas Shift (RWGS) reaction (Equation 2) and the CO Hydrogenation and Boudouard reactions (Equations 3 and 4) favor different catalysts and temperatures to be efficient [5]. This discovery led to the development of Series Bosch (S-Bosch), the system of reactors and filtering membranes shown in Figure 1. S-Bosch premiered the RWGS Reactor (RWGSR) and the Carbon Formation Reactor (CFR) as well as two filtering membranes, Proteus and Polaris, to recycle the unreacted gases into the correct reactor. The RWGSR and membranes have been shown to work in previous testing, but the CFR contains the two remaining issues, catalyst degradation and over-pressurization issues [5], [8], [9], [10], [11], [12].

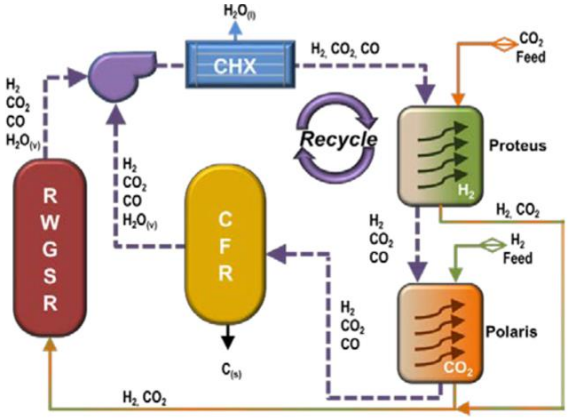


Figure 1. Series Bosch. *A schematic of the S-Bosch test stand consisting of a Reverse Water Gas Shift Reactor (RWGSR), a Carbon Formation Reactor (CFR), the Polaris membrane (depicted in orange), the Proteus membrane (depicted in green), and a Condensing Heat Exchanger (CHX)* **[8]**. *The purple symbol in the top left is a pump.*

Work documented in a previous paper in this series used water to develop a technique to mitigate the reactor over-pressurization issues caused by the formation of carbon clogs as seen in most CFRs. The technique utilized chaotic mechanical agitation in room temperature water to break up the carbon clogs seen in the reactor. This method took one hour to completely break up the carbon clogs for ten grams of reacted material but could be run for longer to remove more of the carbon built up on the bead's surface [13]. The removal of the surface carbon could increase reactor efficiency during cyclical testing when reacting the cleaned beads due to the increase of available activation sites. The catalyst degradation through that cycle was also considered in a previous paper in this series [14]. Efforts to reduce the time necessary for cleaning looked at techniques from the petroleum industry due to Bosch carbon's similarities to coke and the automotive industry where carbon builds up in the pistons and can be removed to increase performance. In the petroleum industry, high velocity steam is injected into pipelines containing coke to thermally shock the catalyst. This allows the carbon to be more easily removed mechanically by a Pipe Inspection Gage (PIG) [15]. Thermal shock also is utilized in the automotive industry to clean carbon deposits by streaming water into a running engine to thermally shock the carbon off the engine block and allow it to come out in the exhaust [16].

In this paper, we will investigate the effects of thermal shock, as seen in the petroleum and automotive industries, on the time necessary for agitation. Since the thermal shock process in an actual CFR will generate saturated steam, testing was done to ensure no additional carbon clogs are generated from interacting with this type of steam by ensuring it did not affect the time needed to agitate the beads. Thermal shock testing entailed quenching reacted material in room temperature Deionized (DI) water to mimic the automotive industry's process and allow for a direct transition to the agitation phase in a reactor. The maximum temperature the beads were heated to before thermally shocking was limited to avoid the formation of a vapor blanket since it creates a different kind of steam and could halt the thermal shock process in zero gravity applications. The Leidenfrost temperature is the temperature where a vapor blanket would form and thus is the limiting factor. It has been found to be between 250 °C and 310 °C for DI water on flat stainless steel [17]. Since the samples are coated in carbon and spherical, 300 °C was chosen as the limit since both attributes increase the Leidenfrost temperature [18] [19]. The minimum desired volumetric contraction was 0.05% leading to a temperature of 42.7 °C. This was found using Equation 5 with β being the linear thermal expansion coefficient for nonresulfurized carbon steel of 8.8×10^{-6}/K, as this was the most representative of the catalyst bead's material, and ΔT being the change in temperature with the lower temperature being room temperature (22 °C) and the higher temperature being 42.7 °C [20]. Using this information, the maximum bead temperature of 300 °C would produce a volume change of 0.73% when quenched in room temperature water.

$$Percent\ Volume\ Change = 3\beta\Delta T \times 100 \tag{5}$$

II. Methods

A gas mixture of carbon monoxide, hydrogen, and nitrogen was fed into the Carbon Dioxide Reduction Catalyst Test Stand (COR-CaTS) reactor to form carbon on the catalysts beads until it reached its pressure threshold due to carbon clogs. A 10 g random sample of the material removed after formation was weighed into either a crucible or a beaker in a nitrogen pressurized glovebox and then underwent one of the processes detailed below. It was then placed into the vacuum chamber attached to the glovebox which was pulled down and pressured with nitrogen three times to ensure air was not entering the glovebox. Mechanical agitation was then performed in the glovebox with 100 mL of room temperature DI water at the highest stir plate setting in a fixed off centered beaker. The contents of the beaker were then strained, and the beaker was rinsed with 25 mL of DI water and poured over the straining material. After straining, the material was placed back into the beaker and heated until dry before getting the final weight. The processes selected for thermal shock and steam testing are detailed below as well as the methods used for mathematical analysis. More details on the carbon formation and mechanical agitation processes are in this series' first paper [12].

A. Saturated Steam Testing

For steam testing, ten grams of reacted material in a 500mL beaker covered loosely with a watch glass underwent a full glassware cycle in a wet steam autoclave (Tuttnauer Brinkmann 3545E) prior to mechanical agitation. During the autoclave cycle, saturated steam was produced by actuation of electrical heaters until a preset temperature of either 105 °C or 134 °C was reached, the temperature was maintained for 99 min, and then the pressure was slowly released over 15 to 20 min until the temperature reached 85 °C to avoid thermally shocking the beads or aerosolizing the loose carbon into the chamber [21]. After the autoclave cycle was completed, the reacted material was either quickly removed with heat protectant gloves or allowed to cool completely in the autoclave then transported to the glovebox.

B. Thermal Shock Testing

Thermal shock testing was conducted at the station depicted in Figure 2. A crucible containing ten grams of reacted material was placed in a nitrogen purged Paragon KMT27 furnace to heat to either 42.7 °C or 300 °C. The nitrogen purged Paragon KMT27 furnace had been preheated to either 46 °C for a test to 42.7 °C or 330 °C for a test to 300 °C. The furnace was being purged with nitrogen to maintain a pressure of 16 psia with a facilities nitrogen pressure of 35 psi. A FLUKE 724 Temperature Calibrator meter attached to an Omega T-type Surface Thermocouple with Self-Adhesive Backing that had been placed in the crucible and surrounded by reacted material was used to determine when the material reached the desired temperature. The crucible was taken out with Blacksmith Depot OC Pickup tongs while wearing heat protectant gloves, the thermocouple was quickly removed, and the reacted material was immediately dumped into a Vollrath 47199 strainer set in a 5 gallon metal bucket that was three quarters of the way full DI water also depicted in Figure 2. The strainer was removed from the water, and the chiseled side of a disposable scoop was used to move the reacted material from the strainer to a 400 mL beaker. The beaker was transported to the mechanical agitation configuration in a Pure Lab HE 2GB glovebox.



Figure 2. Experimental Setup of Thermal Shock Tests.

C. Average Mass Reduction Analysis

The mathematical equations used for establishing the average mass reduction are detailed in Equations 6 and 7. These equations utilized the bead density found a previous paper and sieve opening size information from Amasteel for S660 Cast Steel Shot which was used as the catalyst beads [13]. The variables listed in Equation 6 are as follows: 'm' is average bead mass, 'V' is volume of one bead, 'ρ' is bead density, and 'D' is bead diameter.

$$m = V\rho = \frac{4}{3}\pi\left(\frac{D}{2}\right)^3 * \rho \tag{6}$$

$$\alpha = Average\ mass\ reduction, \% = \frac{mass\ of\ material\ removed}{initial\ sample\ mass - (number\ of\ beads) * average\ bead\ mass} * 100\% \tag{7}$$

III. Results and Discussion

The results were analyzed visually and graphically. Since the elimination of pressure causing clogs was the focus of this study and the main determiner for success, more emphasis was placed on the visual examination of the reacted material after testing to ascertain the existence of pressure causing clogs. Saturated steam and thermal shock were evaluated using that criteria and results are detailed in the appropriate sections below. All figure numbers beginning with A are located in the Appendix starting on page 7.

A. Saturated Steam Testing

Initial testing was performed to determine if the autoclave temperature setting or allowing the beaker to cool completely in the autoclave impacted results. The autoclave temperature was first set to the maximum (134 °C) for one test and the minimum (105 °C) for another test to ascertain if there was a difference. The lower temperature was shown to perform slightly better with less clumps seen when comparing Figure 3ab and Figure A1. The test for the optimal length of time a sample should cool in the autoclave before agitation compared leaving the sample to cool completely to pulling the sample out immediately after the cycle completed. There was no discernable difference between the results as can be seen when comparing Figure 3cd and Figure A1.

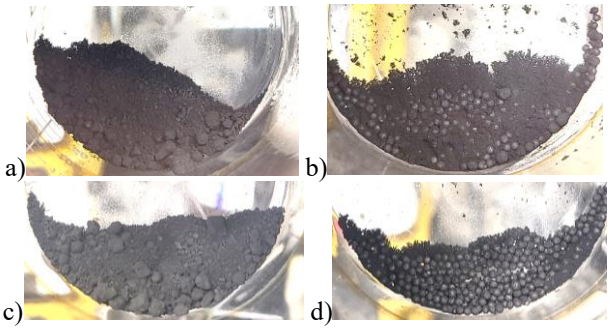


Figure 3. Autoclave Operation Testing. *All images brightened by 30%. a)-b) A before and after image of a catalyst/carbon sample autoclaved at 134 °C and then washed for 1 hr. c)-d) A before and after image of a catalyst/carbon sample autoclaved at 105 °C and allowed to sit until cooled and then washed for 1 hr.*

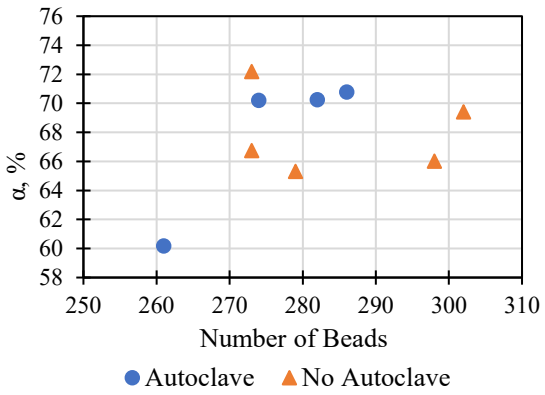


Figure 4. Material Removed from a 45 min Spin after Autoclaving. *In all experiments the autoclave was set to 105 °C, and reacted material was removed directly after the autoclave finished.*

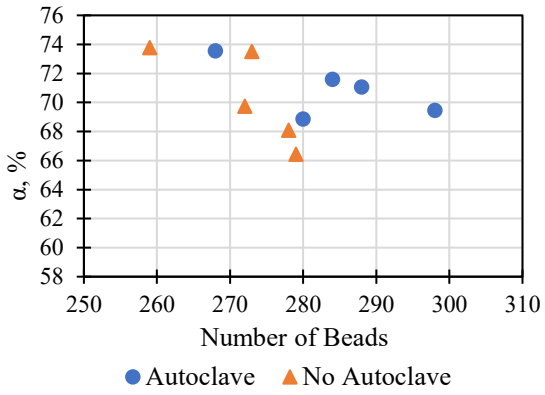


Figure 5. Material Removed from a 1 hr Spin after Autoclaving. *In all experiments the autoclave was set to 105 °C, and reacted material was removed directly after the autoclave finished.*

Repeatability studies were conducted to see if there was a difference in the time necessary to spin after autoclaving with these conditions. It was found that no significant impact was made with the time necessary to break up clumps remaining at 1 hr comparing Figures A1 and A2. There was also no discernable impact on the average mass reduction at either time as seen in Figures 4 and 5. This proves that saturated steam has little to no impact on the effectiveness of agitating the beads and thus will not cause a failure in the washing process if thermal shock is implemented in a reactor design.

B. Thermal Shock

Thermal shock testing occurred at a maximum temperature set to stay below the Leidenfrost temperature and at a temperature predicted to perform approximately a 0.05% volumetric contraction. Those temperatures were 300 °C and 42.7 °C respectively. Testing at the lower temperature revealed that 30 min of agitation was the minimum amount of time necessary to break up the pressure causing clogs as seen when comparing Figure 6 and Figure A3. A minimum of 30 min of agitation was also seen at the higher temperature as seen when comparing Figure 7 and Figure A4.

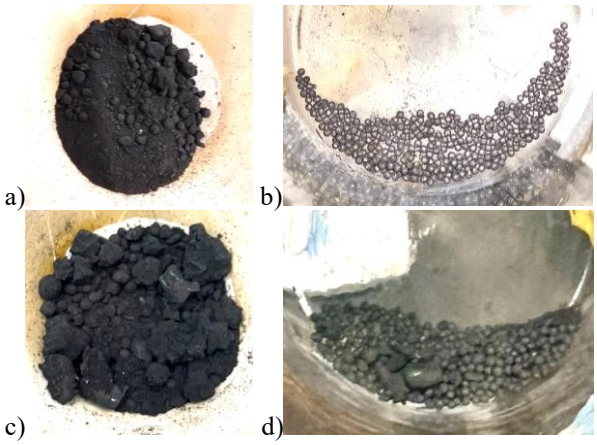


Figure 6. Time Tests for Thermally Shock 42.7 °C. *All images brightened by 30%. a) A representative sample of the material before thermal shock from that day b) A image of the catalyst/carbon sample after being thermally shocked at 42.7 °C and then agitated for 45 min. c)-d) A before and after image of a catalyst/carbon sample thermally shocked at 42.7 °C and then agitated for 15 min.*

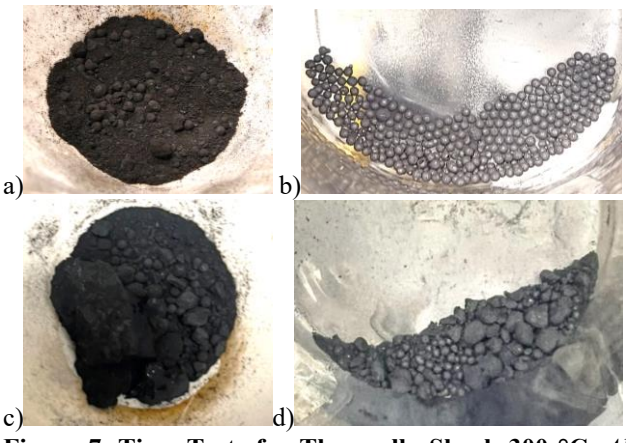


Figure 7. Time Tests for Thermally Shock 300 °C. *All images brightened by 30%. a)-b) A before and after image of a catalyst/carbon sample thermally shocked at 300 °C and then agitated for 45 min. c)-d) A before and after image of a catalyst/carbon sample thermally shocked at 300 °C and then agitated for 15 min.*

Though the clumps were broken up in 30 min at both temperatures, more material was removed from the catalyst beads during the higher thermal shock as seen in Figure 8. The average mass reduction for the higher temperature was the best seen to date from a single cycle in the current configuration for agitation. Due to time restrictions, the investigation into that phenomenon will continue in future work.

These results prove that thermal shock has a positive relationship with decreasing the amount of time the reacted material needed to be agitated, and they show promise for an efficient way to remove the surface carbon with minimal agitation if that is deemed necessary in future cyclical testing. The low temperature and high temperature thermal shocks were both capable of breaking up the pressure causing clogs in a similar time frame. Therefore, a reactor without steam generation is a possibility. This opens an avenue to use this technology on not just a lunar or Martian habitat but also on a space station or a transport vehicle without the need for complex phase separation. This increases the technology's use case and expands the definition of what a viable reactor product could be.

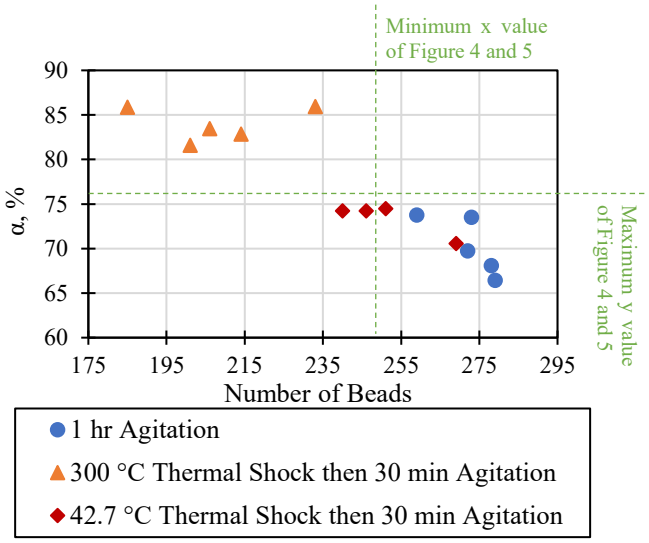


Figure 8. Material Removed from Different Thermal Shock Conditions Compared to the Results without Thermal Shock. *In all experiments, washing conditions were the same. The data for 1 hr agitation comes from a previous paper in this series* **[13]**.

IV. Conclusion and Future Work

The thermal shock trials were very successful, cutting the time necessary for agitation in half, and steam testing showed the agitation process was unaffected by saturated steam. This technology will help propel Bosch to a usable state by opening an avenue to catalyst regeneration with minimal power and time required that will reduce the need for catalyst resupply and thus make Bosch lighter to fly. The next steps to take with this work are more in-depth testing of the correlation between thermal shock and material removed and to run the Bosch process with cleaned beads to find out how the cleaning process affects reactor efficiency. Once that relationship is established, work will turn towards optimizing the relationship between reactor regeneration and operation time. Experiments are currently being conducted to develop lab scale mechanisms to eventually be scaled up and put inside a reactor.

Appendix

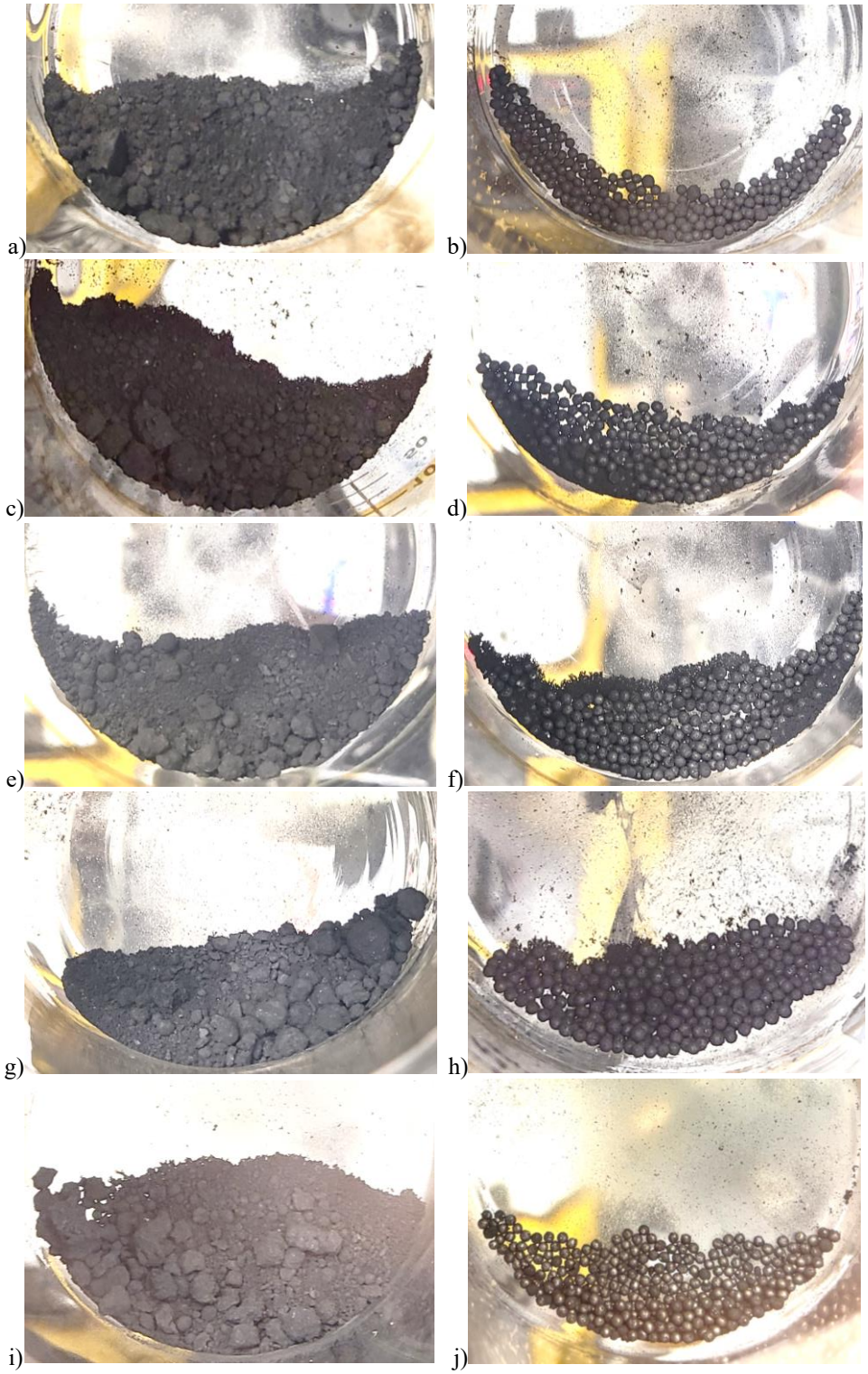


Figure A1. Material Removal from Autoclave and 1 hr Duration Repeatability Experiments. *All images brightened by 30%. a)-b), c)-d), e)-f), g)-h), i)-j) Before and after images of a catalyst/carbon sample autoclaved at 105 °C and then washed for 1 hr.*

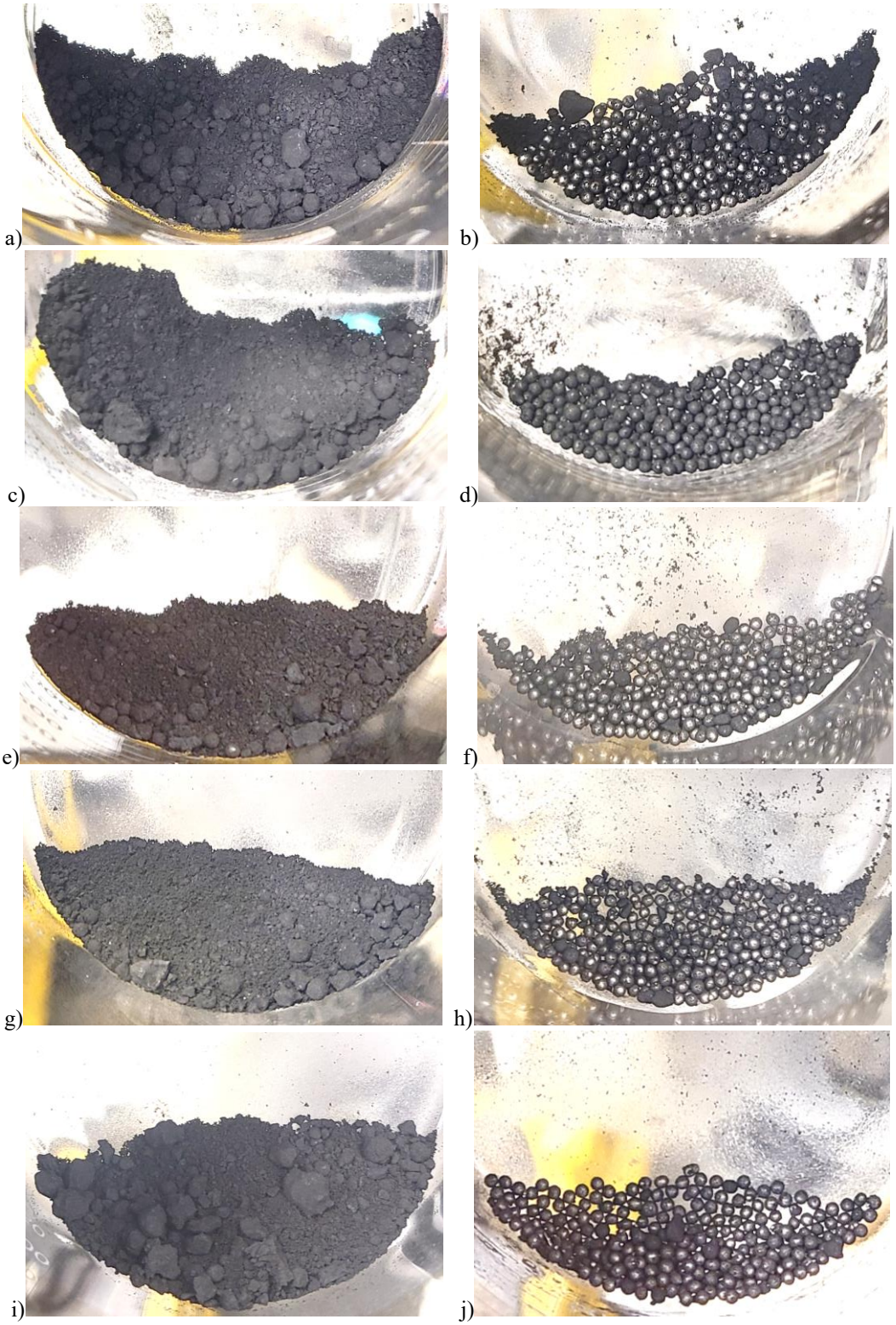


Figure A2. Material Removal from Autoclave and 45 min Duration Repeatability Experiments. *All images brightened by 30%. a)-b), c)-d), e)-f), g)-h), i)-j) Before and after images of a catalyst/carbon sample autoclaved at 105 °C and then washed for 45 min.*

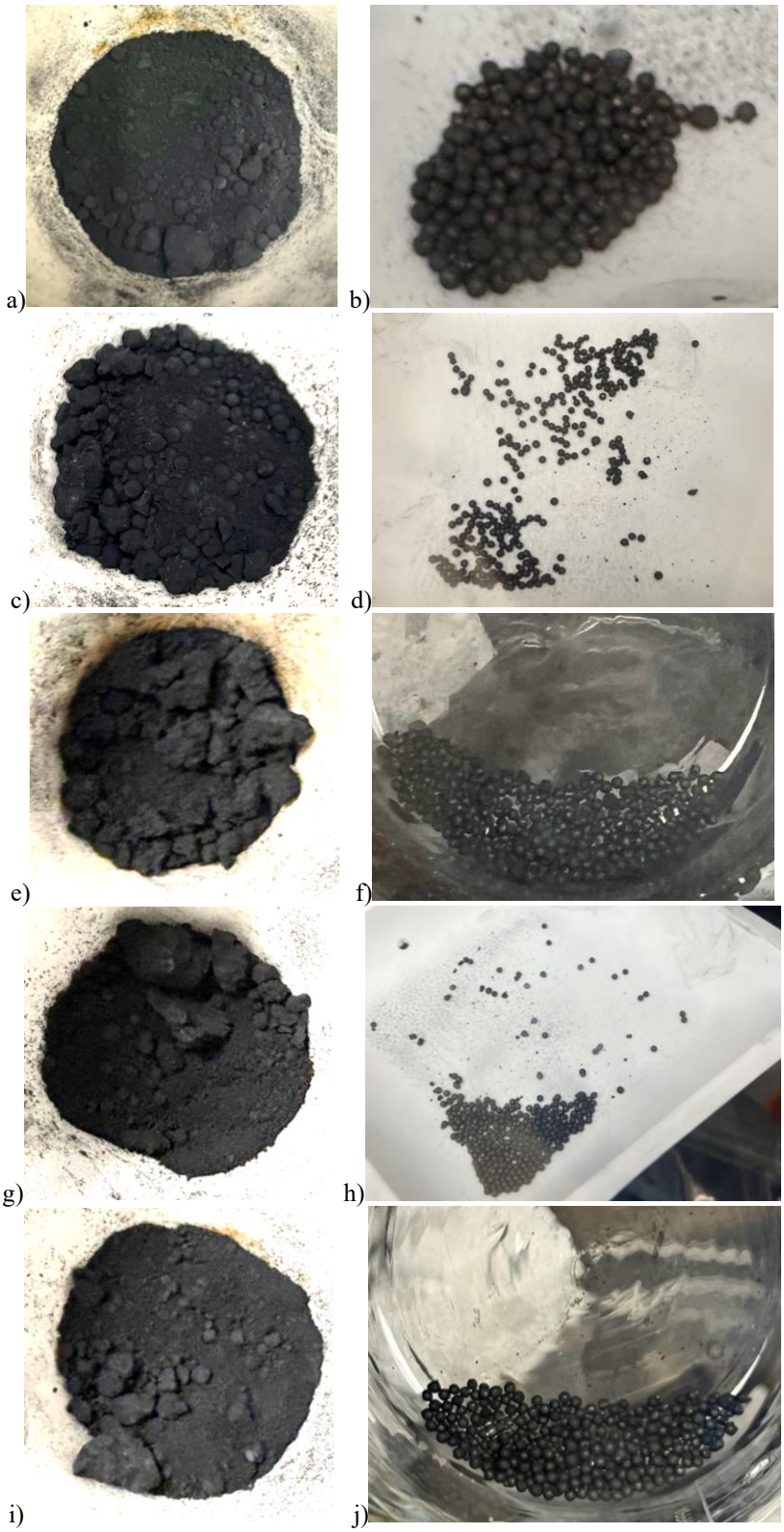


Figure A3. Material Removal from Thermal Shock at 42.7 °C and 30 min Duration Agitation Repeatability Experiments. *All before images brightened by 30%. a)-b), c)-d), e)-f), g)-h), i)-j) Before and after images of a catalyst/carbon sample thermally shocked at 42.7 °C and then washed for 30 min.*

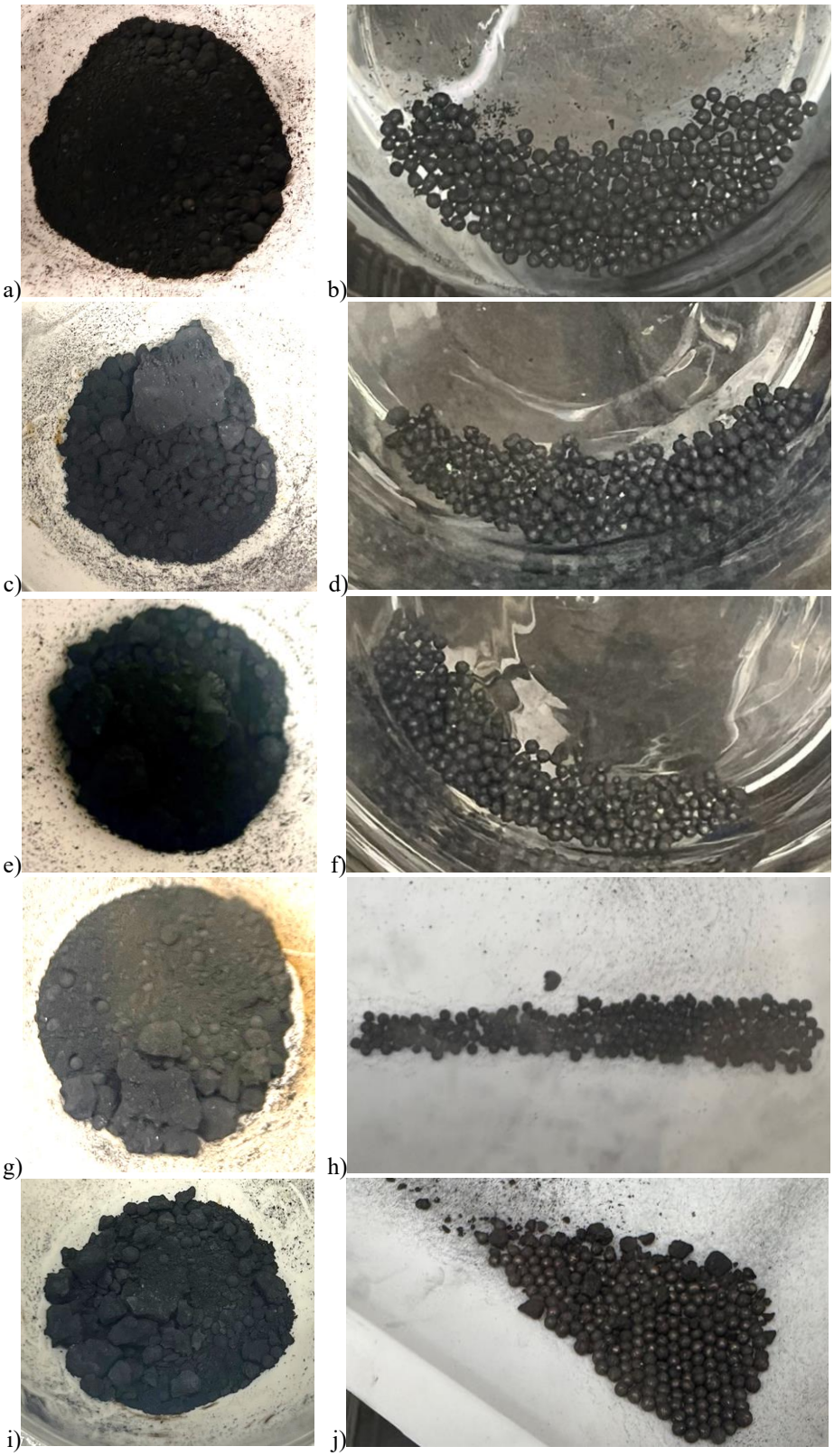


Figure A4. Material Removal from Thermal Shock at 300 °C and 30 min Duration Agitation Repeatability Experiments. *All before images brightened by 30%. a)-b), c)-d), e)-f), g)-h), i)-j) Before and after images of a catalyst/carbon sample thermally shocked at 300 °C and then washed for 30 min.*

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