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Evaluation of iron and cobalt powders as catalysts for ^{14}C -AMS target preparation

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Abstract

We present an evaluation of different brands of cobalt and iron powders used to catalyze CO_2 reduction for ^{14}C -AMS graphite targets at the KCCAMS/University of California, Irvine. The optimal catalyst is characterized by rapid graphitization, homogeneity and lack of sintering of the catalyst/carbon mixture, and contains minimal amounts of both “modern” and “dead” carbon. Fifteen catalyst powders were evaluated using these criteria. The results of this study indicate three good “catalyst-candidates” with backgrounds on processed coal (Argonne Premium coal POC#3) samples around 60 ka BP and modern and dead carbon contamination of $\leq 0.2 \mu\text{gC}$ for 4–5 mg of catalyst.

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Keywords: Radiocarbon; Accelerator mass spectrometry; Catalyst; ^{14}C -AMS sample preparation

1. Introduction

In most ^{14}C -AMS laboratories, carbon is chemically separated and purified from the original sample as CO_2 and catalytically reduced to graphite. Then, it is pressed and loaded as a target for the sputter ion source, where it is converted into a beam of charged particles. There are some well known procedures available to produce graphite targets [1–4]. Over the past two decades, many approaches have been taken by researchers to quantify and minimize contamination effects resulting from these sample preparation protocols [5–9] as well as to improve the yield and quality of the graphite produced [10–13]. An important factor in the catalytic reduction reaction overlooked in previous studies is the catalyst itself. In this work, we evaluate the purity and behavior of the manufactured catalysts com-

monly used in ^{14}C -AMS sample preparation. We believe they play an important role in the graphitization process, particularly for samples in the microgram range, where both “modern” and “dead” contamination are major concerns [14–16,13,17]. We determine the suitability of one cobalt and 14 iron catalysts for graphite production. Our primary goal was to find a catalyst which produces high quality graphite rapidly and with high conversion efficiency, allowing large batches of samples to be processed with reproducible quality, and which could be used with minimal improvement of the “off the shelf” product.

2. Experiments and results

Fifteen catalysts were tested in this work. The selection of Fe and Co powders was based on the manufacturing process of the catalyst (carbonyl, electrolytic, reduced), mesh size and purity level as reported by the producer (97–99.9% pure). Several of these catalysts are used by other laboratories [11–13]. Table 1 shows all catalysts evaluated and the information reported by the manufactures.

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Table 1
Catalyst brands investigated in this study

Catalyst #	Company name	Mesh	Name	Purity (%)	Batch lot #
1	CERAC	–100	Iron metal, Fe	99.9	X25213-112602
2	CERAC	–325	Iron metal, Fe	99.9	X0027677-1
3	Sigma–Aldrich	–325	Iron, powder	97.0	03408 PA
4	Sigma–Aldrich	–400	Iron, powder	99.9	05406 EA
5	Alfa–Aesar	–200	Iron, powder	99.0	737
6	Alfa–Aesar	–325	Iron, powder	98.0	J02M27
7	AEE	–100	FE-103 iron powder (electrolytic)	99.9	–
8	AEE	–100/+325	FE-112 iron powder (H ₂ reduced)	99.8	408536
9	AEE	–325	FE-102 iron powder (electrolytic)	99.9	404500
10	AEE	–325	FE-110 iron powder (H ₂ reduced)	99.8	2224
11	NOAH	–325	Iron metal, powder reduced	99.95	139734/3.1
12	NOAH	–325	Iron metal, powder electrolytic	99.9	67394/0.0
13	Alfa–Aesar	–300	Cobalt powder	99.8	A27BO2
14	ISP	–200	Iron, powder – GRADE R2140	–	–
15	Hoganas	–325	Iron, powder – GRADE A-230	>98.5	4504053

Table shows general information reported by the producers (company name, mesh size, catalyst name, reagent grade, purity level for other metals and elements, batch or lot number).

Legend: AEE: Atlantic Equipment Engineers; NOAH Tech. Corp.: NOAH Technologies Corporation; ISP: International Specialty Products; Hoganas: North America Hoganas/High Alloys LLC.

Each catalyst is identified by a catalyst number from 1 to 15 for easy reference, company name and mesh size.

To evaluate the suitability of the catalyst used to produce graphite targets for ¹⁴C-AMS measurements, we used the criteria below:

- ¹²C⁺¹ beam currents from catalyst pressed directly into sample holders were measured to evaluate the carbon content of the catalysts before and after pre-baking (400 °C for 1 h in 1 atmosphere of H₂). ¹⁴C/¹²C ratios were also measured for the ion beam currents from the untreated and pre-baked catalyst.
- Blank values for coal (Argonne Premium Coal POC#3) samples produced with these catalysts (~1 mgC graphite per 4–5 mg of catalyst) were determined.
- We measured the conversion time of CO₂ to graphite for these catalysts.
- We assessed the homogeneity and lack of sintering of the catalyst/carbon mixture.
- We evaluated the ¹²C⁺¹ beam current intensity produced from the graphite targets.

Catalysts which were found unsuitable during initial tests were eliminated from further consideration.

2.1. ¹²C⁺¹ beam currents and ¹⁴C/¹²C ratios from untreated and pre-baked catalyst (criterion 1)

To investigate the carbon deposited on or in the catalyst grains, we performed ¹⁴C-AMS measurements on untreated and pre-baked catalyst powder pressed into our aluminum cathode holders. We tuned the entire AMS system with a set of 1 mgC graphite samples of standard reference materials (such as NBS Oxalic Acid I (OX-I) and NBS Oxalic Acid II SRM 4990-C (OX-II)) to assure the correct settings for ¹⁴C measurements. Then, we adjusted

the range of the current measurement system to record the very low beam currents expected from the catalyst targets. For each catalyst, at least two cathode holders for both untreated and reduced catalyst were tested. Carbon beam currents produced from empty holders were a negligible fraction (~2%) of those from catalysts.

Most of the catalysts investigated by the procedure describe above gave similar beam currents (within ±30%) indicating comparable carbon content. Beam currents varied from day to day, but were typically 0.3% of those from 1 mgC graphite samples and ¹⁴C/C ratios were in the range of 0.5–1 times modern. The irons FE-103 Electrolytic –100 mesh and FE-110 hydrogen reduced –325 mesh from Atlantic Equipment Engineers (Table 1) were cut for further investigations based on their carbon impurity levels observed during this experiment (exceptionally high compare with others). The cobalt Alfa–Aesar –300 mesh catalyst was also clearly contaminated at the particulate level since the beam current intensity was high and erratic. However, we continue to test this catalyst, since it was used in another sample preparation laboratory at UCI.

2.2. Overall performance of graphite/catalyst mixtures (criteria 2 to 5)

For testing criteria 2–5, at least three H₂ reduced graphite targets were produced per catalyst according to the laboratory protocol described in Santos et al. [18]. The “¹⁴C-dead” reference material selected for these experiments was coal (Argonne Premium Coal POC#3) chemically pre-treated by the conventional acid–alkaline–acid (AAA) procedure.

2.2.1. Blank values

The UCI KCCAMS prep-laboratory is equipped with two identical graphitization lines each having 12H₂/Fe reduction reactors. Prior to graphitization each reactor

was loaded with a different catalyst and all of them were pre-baked at 400 °C with ~1 atmosphere of H₂. A large single pool of CO₂ was then divided into 11 samples and reduced to graphite. Note that two catalysts were eliminated by criterion 1. Two extra catalysts were added to the set later and will be addressed below. Enough CO₂ was transferred to each graphitization reactor to produce ~1 mgC graphite sample. We repeated this process three times using the same vacuum line. The ¹⁴C-AMS results from the set of 11 catalysts are shown in Table 2. Note that the first reported result from each catalyst (Table 2 – batch 1) is slightly younger, when compared with the second and third results (batches 2 and 3). This first result shows a small memory effect in our graphitization reactors [19]. Two additional iron catalysts ISP R2140 –200 mesh and Hoganas –325 mesh were obtained later and evaluated separately, using some of the catalysts tested previously as controls. The ¹⁴C-AMS results from this second set of mea-

surements are shown in Table 3. Four graphite targets per catalyst were produced in the same fashion as the first set. However, we measured only the last three targets because of the memory effect: We believe that the ¹⁴C-AMS result from the first target produced, if measured, does not reflect the true blank that can be achieved by the catalyst investigated. There was no overall difference in the background values between all catalysts tested (average ages are ~60 ka BP – Table 2 and 3), except for the cobalt Alfa-Aesar –300 mesh as expected due to its high carbon content (criterion 1).

2.2.2. Reduction time

The iron powders Sigma-Aldrich –400 mesh, Alfa-Aesar –325 mesh, NOAH –325 mesh reduced and ISP R2140 –200 mesh show an average reduction time of ~120 min (Fig. 1) which is the shortest among the 15 catalysts evaluated. Note that no differences from the usual

Table 2
Fraction modern values and ¹⁴C ages from AMS measurements on coal samples (first measured set of catalysts)

UCIAMS #	Batch #	Ident. (name)	Fract. mod	± (1δ)	¹⁴ C Age (yrs. BP)	± (1δ)	Physical assessment	
							Homogenous	Texture
15032	1	Fe # 1; CERAC; –100	0.0008	4.2E–05	57500	440	Yes	Fluffy
15033	2		0.0007	3.5E–05	58550	420		
15034	3		0.0004	2.6E–05	62660	510		
15035	1	Fe # 2; CERAC; –325	0.0010	4.6E–05	55430	370	Yes	Fluffy
15036	2		0.0005	3.3E–05	60620	500		
15037	3		0.0005	2.6E–05	61030	430		
15038	1	Fe # 3; Sigma-Aldrich; –325	0.0013	7.5E–05	53480	470	Yes	Fluffy
15039	2		0.0005	2.6E–05	60980	410		
15040	3		0.0004	2.7E–05	62440	530		
15041	1	Fe # 4; Sigma-Aldrich; –400	0.0007	3.9E–05	58210	450	No	Solid
15042	2		0.0004	2.1E–05	63010	440		
15043	3		0.0004	2.4E–05	62620	480		
15044	1	Fe # 5; Alfa-Aesar; –200	0.0010	5.2E–05	55720	440	Yes	Fluffy
15045	2		0.0005	2.5E–05	60780	390		
15046	3		0.0004	2.6E–05	61990	470		
15028	1	Fe # 6; Alfa-Aesar; –325	0.0007	3.7E–05	58040	410	No	Fluffy
15030	2		0.0006	2.5E–05	59850	360		
15031	3		0.0005	3.5E–05	61270	580		
15047	1	Fe # 8; AEE; –100/+325	0.0010	4.8E–05	55590	400	Yes	Firm
15048	2		0.0005	2.4E–05	60790	380		
15049	3		0.0005	3.9E–05	61220	640		
15050	1	Fe # 9; AEE; –325	0.0012	5.0E–05	54110	350	Yes	Fluffy
15051	2		0.0005	2.3E–05	61610	400		
15052	3		0.0004	2.6E–05	61930	470		
15053	1	Fe # 11; NOAH; –325; Reduced	0.0009	3.8E–05	56090	340	Yes	Fluffy
15054	2		0.0006	2.8E–05	59740	380		
15055	3		0.0004	2.6E–05	62260	480		
15056	1	Fe # 12; NOAH; –325; Electrolytic	0.0009	3.8E–05	56640	360	Yes	Fluffy
15057	2		0.0005	3.2E–05	60440	480		
15058	3		0.0004	3.4E–05	62580	660		
15059	1	Co #13; Alfa-Aesar; –300	0.0012	4.7E–05	54330	330	Yes	Fluffy
15060	2		0.0010	5.2E–05	55230	410		
15061	3		0.0009	5.7E–05	56460	520		

Samples were graphitized consecutively in the same line to produce three replicates per catalyst. Samples were also assessed during pressing for homogeneity and lack of sintering.

Table 3
Fraction modern values and ^{14}C ages from AMS measurements on coal samples (second measured set of catalysts)

UCIAMS #	Ident. (name)	Fract. mod	$\pm (\delta)$	^{14}C Age (yrs. BP)	$\pm (\delta)$	Physical assessment	
						Homogenous	Texture
15704	Fe # 4; Sigma-Aldrich; –400	0.0006	2.7E–05	59670	380	No	Solid
15705		0.0006	2.6E–05	59770	360		
15706		0.0004	2.5E–05	62150	470		
15709	Fe # 6; Alfa-Aesar; –325	0.0006	3.0E–05	59380	390	No	Fluffy
15710		0.0006	2.4E–05	59600	330		
15711		0.0007	2.5E–05	58140	290		
15714	Fe # 11; NOAH; –325; Reduced	0.0006	3.0E–05	59190	390	Yes	Fluffy
15715		0.0005	2.4E–05	61140	390		
15716		0.0005	2.2E–05	60850	350		
15719	Fe # 14; ISP R2140; –200	0.0006	3.6E–05	59260	470	Yes	Solid
15720		0.0006	3.7E–05	59020	470		
15721		0.0006	2.4E–05	60180	350		
17213	Fe # 15; Hogan A-230; –325	0.0009	3.6E–05	56090	320	Yes	Fluffy
17214		0.0008	2.6E–05	56860	250		
17215		0.0008	2.6E–05	57320	260		

We use the irons Sigma-Aldrich –400 mesh, Alfa-Aesar –325 mesh, NOAH –325 mesh reduced as controls to assess the reduction rate and background levels of the irons ISP R2140 –200 mesh and Hogan A-230 –325 mesh. Samples were also assessed during pressing for homogeneity and lack of sintering.

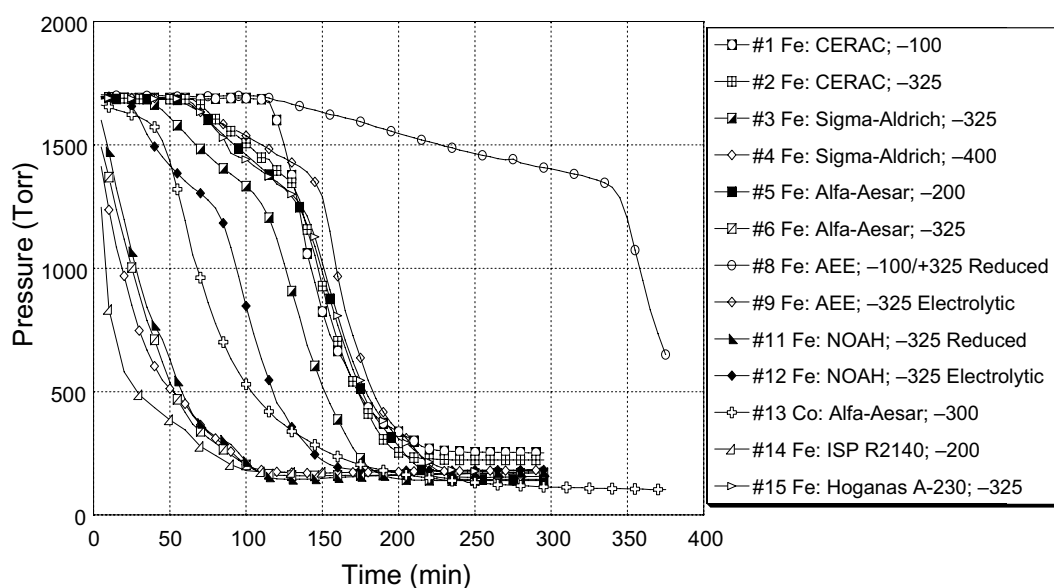


Fig. 1. Reduction rate curve represented by the change in total pressure ($\text{CO}_2 + \text{H}_2$) as a function of time during the catalytic reduction of 1 mgC graphite coal samples by independent catalysts. The reduction process was monitored by recording the pressure drop in each reactor every five minutes until the reaction reached completion. The plot was adjusted to reduce points displayed to better show details on CO_2 reduction time of different catalysts.

protocol [18] were applied to better activate these iron catalysts. A further decrease in reduction time may or may not be possible for these particular iron powders, if different approaches to activate the catalyst are applied [13,20]. Further investigations should be done in this case.

2.2.3. Homogeneity and lack of sintering

Our cathode holders are 6.35×12.7 mm cylinders of Al with a front conical depression [21]. Graphite is loaded from the front of the sample holder by tamping it into the central 1 mm hole, then pressed by hammering on a piece of drill rod. To minimize cleaning time during the pressing proce-

duce, we do not use any extra apparatus such as funnels to contain the graphite on the front of the holder.

The homogeneity of the graphite was evaluated by observation of each graphite target through a microscope (10 \times to 40 \times power range) prior to pressing. Graphite targets were considered homogeneous when the graphite produced was black with little or no definition between carbon and catalyst spots and non-homogeneous when it was black/charcoal/grey with visible catalyst-granular spots.

The physical characteristics of the final carbon/catalyst mixture also play an important role to determine which catalyst provides final graphite targets that minimize press-

ing time and material losses. The extent of sintering was assessed from the catalyst/carbon mixtures, where a criterion to characterize the texture and hardness of graphite pellets was defined as:

- A. Fluffy: Carbon/catalyst mixture falls easily into the target holder as powder and can be easily compressed. Carbon/catalyst mixture does not stick to inside walls of the borosilicate graphitization tube;
- B. Firm: Only some of the mixture falls easily into target holder. Some graphite is stuck to the wall of the tube. It is possible that some graphite was lost due to static

or other effects. Most of the mixture is in the form of a pellet that can be easily broken up by hand with a pressing pin;

- C. Solid: Most of the graphite material is sintered into a pellet and/or stuck to the wall of the tube. It is usually necessary to use a spatula to remove the material on the wall. The mixture is very hard and is difficult to break apart with the pressing-pin in order to get it into the target holder.

Complete assessment for all catalysts studied is this work can be also seen in Tables 2 and 3.

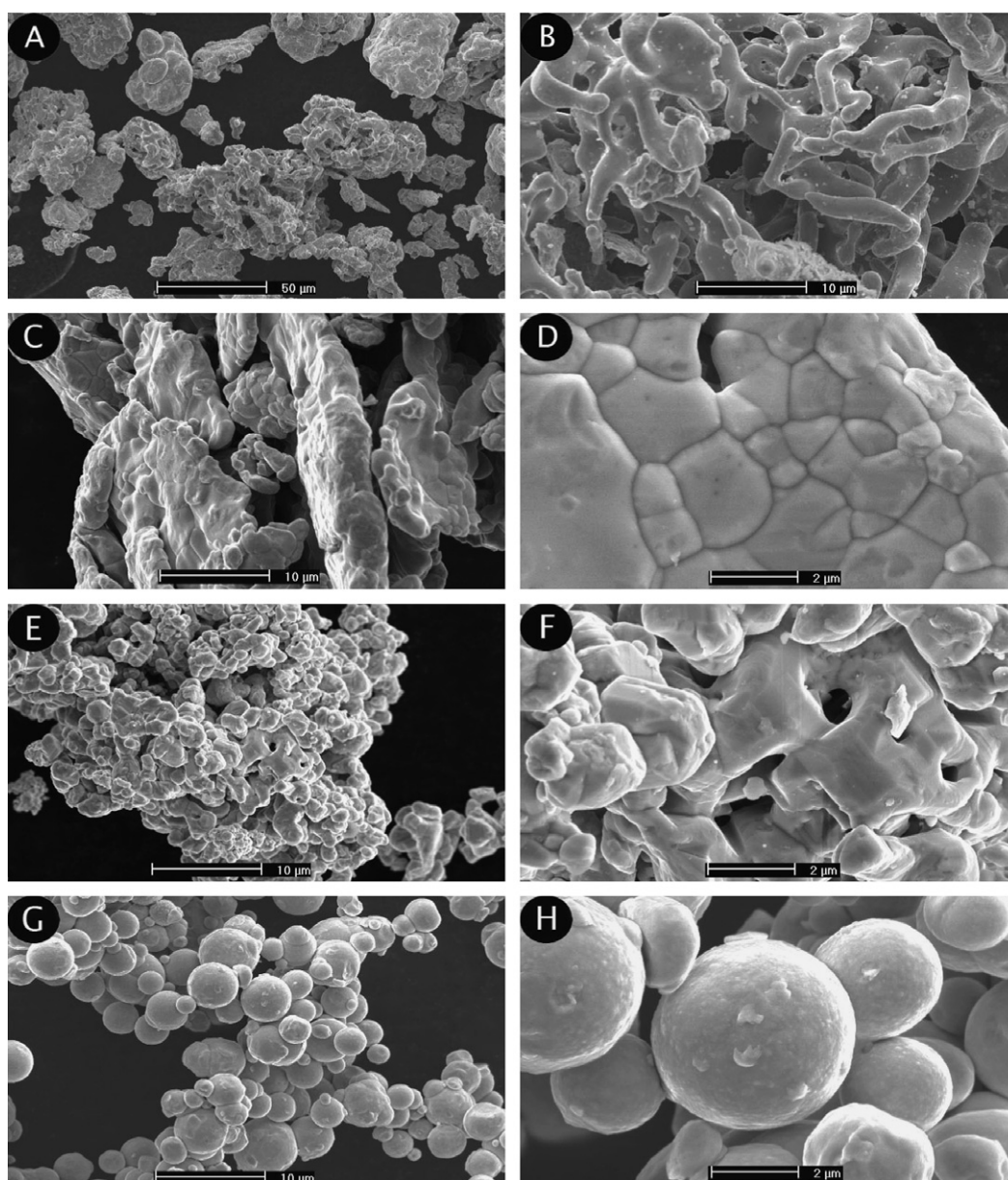


Fig. 2. SEM pictures from some of the catalysts studied in this paper: (A and B) Iron Alfa-Aesar –325 mesh, 50 μm and 10 μm , respectively; (C and D) Iron CERAC –325 mesh, 10 μm and 2 μm , respectively; (E and F) Cobalt Alfa-Aesar –300 mesh, 10 μm and 2 μm , respectively; (G and H) Iron Sigma-Aldrich –400 mesh, 10 μm and 2 μm , respectively.

2.2.4. C^- beam currents

Three wheels were measured for this work and the $^{12}C^1$ beam current produced from the graphite targets evaluated against each other. Overall $^{12}C^-$ beam currents ranged from 90 to 120 μA among all catalyst studied, with catalysts Sigma–Aldrich –400 mesh and Alfa–Aesar –325 mesh yielding the highest currents and Co Alfa–Aesar –300 mesh the lowest.

3. SEM pictures

Filamentous graphite (carbon nanofibers) is found in many forms and configurations. Structures formed depend

on many drivers: thermodynamics parameters, metal catalysts used, size of particle and catalyst surface morphology are some of them. Before filament growth, it is believed that the surface of catalyst particles is transformed to iron carbide (Fe_3C), which cracks and breaks up into sub-micron particles. Carbon reduced on the leading face of these particles diffuses through them and precipitates at the rear face, producing the filament [22,23].

The structure of each untreated catalyst before and after pre-baking was investigated by Scanning Electron Microscopic (SEM) analysis taken at the Materials Characterization Facility, UCI, using a Schottky thermal field emission FEI/Philips XL-30 SEM with back scattered electron

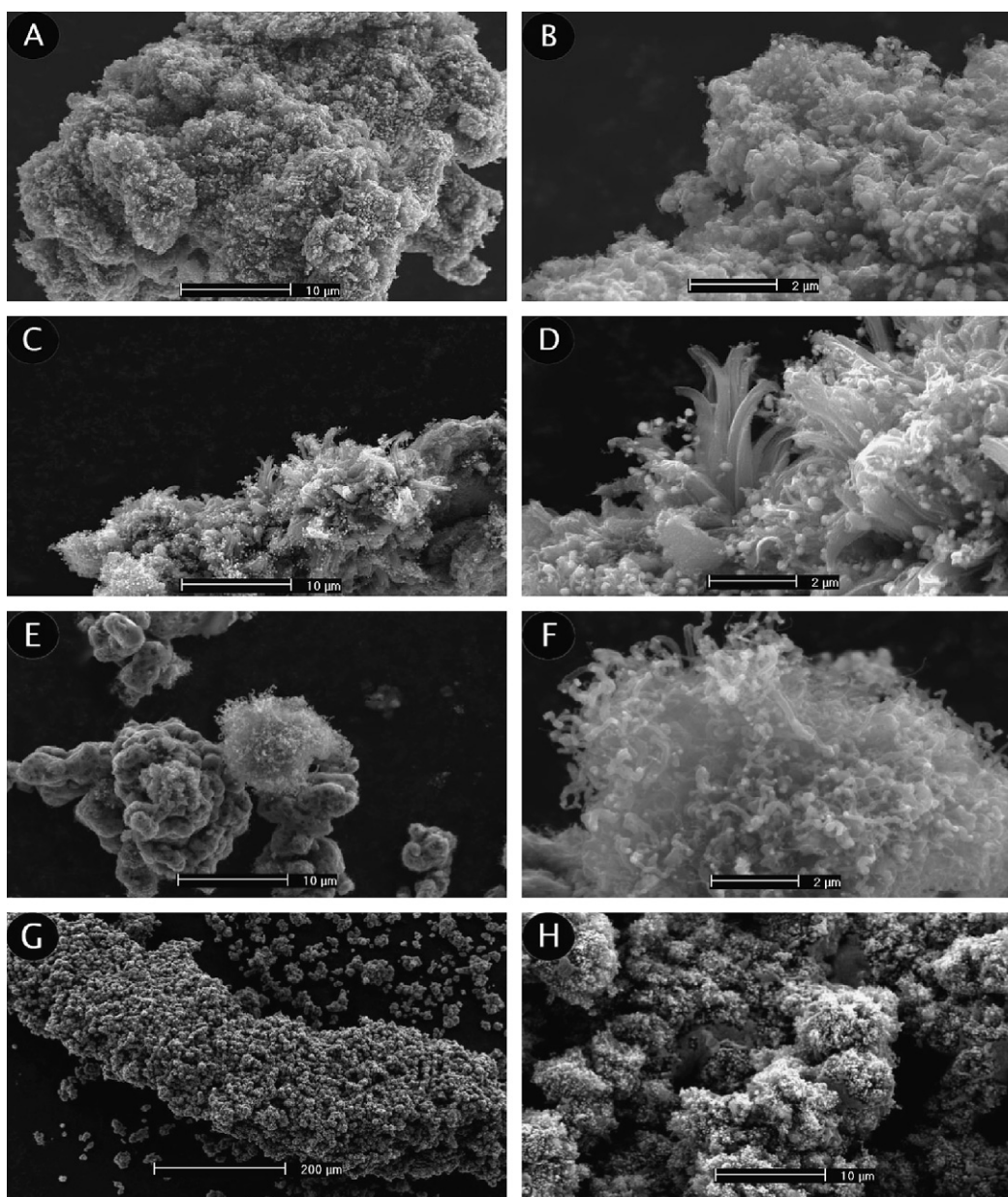


Fig. 3. SEM pictures from some of the graphite samples produced from AAA pre-treated coal CO_2 cryogenically clean gas using different catalysts: (A and B) Iron Alfa–Aesar –325 mesh, 10 μm and 2 μm , respectively; (C and D) Iron CERAC –325 mesh, 10 μm and 2 μm , respectively; (E and F) Cobalt Alfa–Aesar –300 mesh, 10 μm and 2 μm , respectively; (G and H) Iron Sigma–Aldrich –400 mesh, 200 μm and 10 μm , respectively.

detector. The SEM pictures show some similarities for many of the catalysts analyzed, such as coarse fragments and small surface areas. In addition, there is no obvious physical difference between untreated and pre-baked catalysts (Fig. 2(A)–(F)). The carbonyl irons Sigma–Aldrich –400 mesh (Fig. 2(G) and (H)) and ISP R2140 –200 mesh are an exception: these consist of aggregates of small spherical particles with a large surface area.

An independent set of graphite targets were produced for each catalyst for SEM pictures. All graphite produced shows filamentous graphite growth (Fig. 3), however some looks patchier than others. At high magnifications, small catalyst particles are visible on the tips of many of the graphite filaments. As mentioned above, the untreated irons Sigma–Aldrich –400 mesh (Fig. 2(G) and (H)) and ISP R2140 –200 mesh have almost indistinguishable morphologies. Both were strongly sintered during graphitization (Tables 2 and 3). However, the filamentous graphite produced from the two catalysts is very different in shape: ISP R2140 –200 mesh produces structures covered by very fine filaments, when Sigma–Aldrich –400 mesh (Fig. 3(G) and (H)) produces large solid looking structures.

4. Conclusions

We determined that irons Sigma–Aldrich –325 mesh, Alfa–Aesar –325 mesh and NOAH –325 mesh reduced adequately fulfilled the criteria listed. Following this investigation, we replaced the iron catalyst Sigma–Aldrich –400 mesh by Alfa–Aesar –325 mesh because it made pressing procedure easier, without substantially affecting C^- beam current intensity and background levels. The iron Sigma–Aldrich, –400 mesh was discarded due to sintering, despite slightly better backgrounds (Table 1). ^{14}C -AMS measurement results on graphite samples using the new catalyst showed that overall precision (0.2–0.3%) was not affected.

Choosing an appropriate catalyst can clearly decrease background levels and also reduce graphitization and pressing time. Based on this study, another sample preparation laboratory at UCI also switched from Alfa–Aesar –300 mesh cobalt to Alfa–Aesar –325 mesh iron catalyst and it demonstrated improved performance with the iron [24]. However, because graphitization techniques and requirements differ, systematic testing as discussed here may be required to determine the optimum catalyst for other laboratories. Furthermore, it is important to understand that this kind of evaluation may need to be repeated for any particular powder because there is no guarantee that manufacturing processes will remain the same. As a result, powder from the same manufacturer may differ in catalytic properties from one lot to the next.

Note added after peer review of the manuscript

After the submission of this paper for peer review, the Fe powder Alfa–Aesar –325 mesh Lot# JO2M27 was sent

to the Center for Accelerator Mass Spectrometry (CAMS) at LLNL for evaluation. Twelve targets loaded with pure iron were prepared. The targets produced enough lithium to “light up” the detector, showing that this particular Lot# is unsuitable for production of graphite targets for measurement with charge state +4. The authors decided to add this brief note to the paper in order to inform the radiocarbon community of this new information.

Two other Lot#s CO2PO1 and L16P22, also from Alfa–Aesar –325 mesh size, were also evaluated by both CAMS/LLNL and KCCAMS/UCI. At the KCCAMS facility (which operates a 500 kV compact AMS unit from National Electrostatics Corporation), the irons were used to produce regular graphite targets (1 mgC) and sub-milligram targets of ~ 0.025 mgC and ~ 0.006 mgC (three graphite samples per mgC size and Fe powder Lot#) of IAEA-C8 standard. All graphite samples were reduced using our procedural protocols [17,18] with CO_2 reduction reaction yield close to 100%. Subsequently, these graphite targets were measured on a wheel containing sub-milligram targets produced by the Fe powder Alfa–Aesar –325 mesh Lot# JO2M27 as controls. There was no overall difference in the radiocarbon results between all catalysts tested, showing that all three Lot#s (JO2M27, CO2PO1 and L16P22) are suitable to produce graphite targets if measured by particle accelerators not set up for 4+ charge state. At the CAMS/LLNL, the Fe powders Alfa–Aesar –325 mesh Lot#s L16P22 was the only one found to have lower lithium content. Now, CAMS/LLNL is using this Fe powder to produce their graphite targets.

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