

2004

Development, Fabrication and Study of Fullerene-Containing Carbon Material (FCC) for Immobilization of Iodine: Final Report #1

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Savopulo, M., Burakov, B. E. (2004). Development, Fabrication and Study of Fullerene-Containing Carbon Material (FCC) for Immobilization of Iodine: Final Report #1. 1-6.

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Research-Industrial Enterprise “KIRSI”, branch of the V.G. Khlopin Radium Institute (KRI)

UNLV – KRI-KIRSI Agreement 280203-1

**Development, Fabrication and Study of Fullerene-Containing
Carbon Material (FCC) for Immobilization of Iodine**

Progress Reports #1-4

Examination of main features
of iodine-doped Si_xC_y

Saint-Petersburg - 2005

1. Introduction

During current reporting period the experiments on synthesis of ceramic-like material by conversion of iodine-doped FCC and activated carbon to Si_xC_y have been completed. Cold pressed granules of FCC and activated carbon were rinsed in water-ethanol solution of tetraethoxysilane (TEOS), $\text{Si}(\text{OC}_2\text{H}_5)_4$, and then used for further synthesis. All new samples obtained have been studied using precise powder XRD analysis.

2. FCC samples

- 1) Initial FCC containing 6.2 wt.% fullerenes. This material was used for all other experiments.
- 2) FCC + TEOS dried under infra-red lamp in air for 1 hour.
- 3) Sample #2 after sintering in vacuum at 800 °C for 30 minutes.
- 4) Sample #2 after affecting by laser (with beam diameter 1 mm and energy 10 joules) at both pellet sides.
- 5) FCC doped with iodine (252 mg/g).
- 6) Iodine doped FCC (sample #5) + TEOS dried under infra-red lamp in air for 1 hour.
- 7) Samples #6 after sintering in vacuum at 400 °C for 30 minutes.
- 8) Samples #6 after affecting by laser (with beam diameter 1 mm and energy 10 joules) at both pellet sides.

3. Properties of FCC interacted with Si-organics

The results of XRD analyses are summarized in Figures 1 and 2. Initial undoped FCC sample (Fig.1, spectrum #1) consists of three phases: amorphous carbon (see typical broad band in low angle area up to 30 degrees); graphite and fullerenes (mainly C_{60}). Low temperature interaction between FCC and Si-organics (Fig.1, spectrum #2) caused change of XRD spectrum in the area of amorphous carbon what means as assumed the formation of new unknown amorphous phase. In the same time decrease of C_{60} content was observed. Further sintering of this sample in vacuum (Fig.1, spectrum #3) did not cause formation of crystalline Si_xC_y phase; however, it recovered the shape of XRD spectrum in low angle area. Graphite content increased in comparison with initial FCC up to 50-60 %. Our interpretation is that under vacuum conditions at 800 °C amorphous Si-phase evaporated but it should be confirmed by chemical analysis. Very interesting result was obtained after affecting altered FCC sample (interacted with Si-organics under infra-red lamp) by laser beam in air (Fig.1, spectrum #4). Intensity of C_{60} peaks increased, however, the shape of XRD spectrum in low angle area remained the same as before laser affect. This means that laser helps formation of new fullerenes as well as it does not destroy new-formed amorphous unknown phase (probably, Si-C-phase). This assumption correlates with essential decrease of graphite content – up to 40 % in comparison with initial FCC.

Current experiments on iodine doping completely confirmed our previous results that iodine actively interact with FCC. This interaction is accompanied with partial destruction of graphite and fullerenes and formation of carbyne chains (Fig.2, spectrum #5, low angle area). Interaction of iodine doped FCC with Si-organics in air at low temperature (Fig.2, spectrum #6) and under vacuum conditions at 400 °C (Fig.2, spectrum #7) caused increase of graphite content. After sintering in vacuum increase of graphite was about 50 %, which is the same as for undoped FCC (Fig.1, spectrum #3).

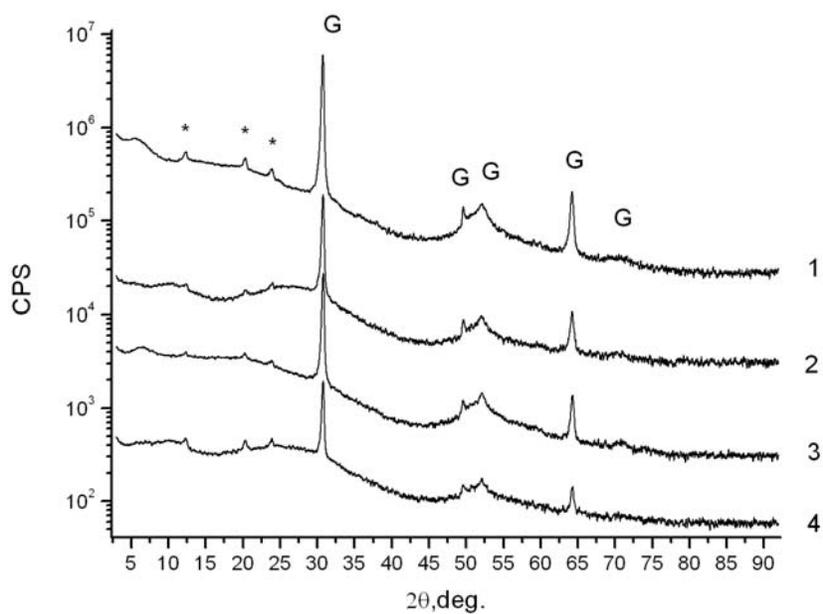


Figure 1. XRD patterns of **undoped FCC**: (1)- initial and (2-4) after interaction with Si-organics under different conditions. Peaks of graphite are marked as “G” and C_{60} as *.

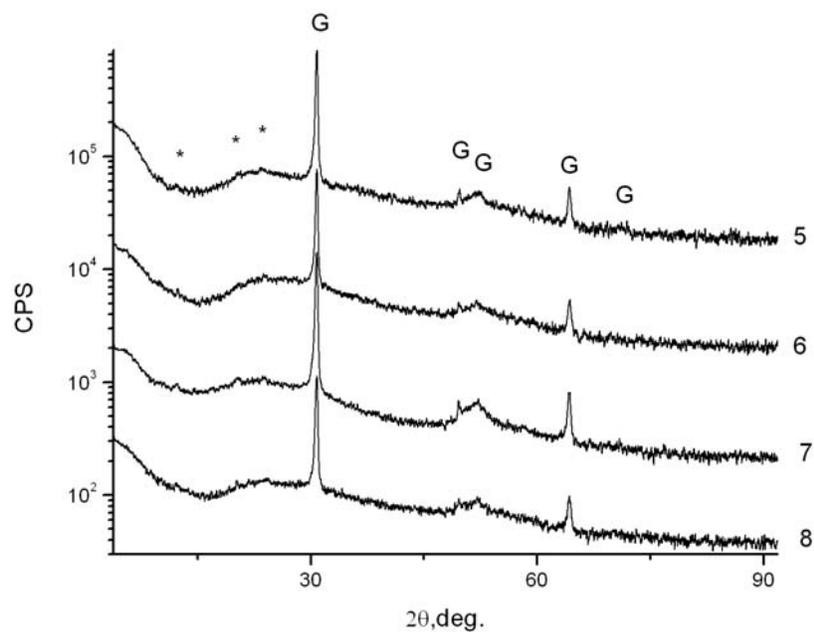


Figure 2. XRD patterns of **iodine doped FCC**: (5)- initial and (6-8) after interaction with Si-organics under different conditions. Peaks of graphite are marked as “G” and C_{60} as *.

The behavior of iodine doped FCC (altered by Si-organics) under laser beam (Fig.2, spectrum #8) is similar to undoped FCC: there is no change of XRD spectrum in low angle area (therefore, no destruction of new formed unknown amorphous phase, possibly, Si-C) and decrease of graphite content up to 40 % in comparison with initial iodine doped FCC (although, formation of new fullerenes was not clear confirmed).

4. Samples of activated carbon

- 1) Initial activated carbon SKT-3S using as an absorbent in Russian nuclear industry.
- 2) Activated carbon + TEOS dried under infra-red lamp in air for 1 hour.
- 3) Sample #2 after sintering in vacuum at 800 °C for 30 minutes.
- 4) Activated carbon SKT-3S doped with iodine (57 mg/g).
- 5) Iodine doped activated carbon (sample #4) + TEOS dried under infra-red lamp in air for 1 hour.
- 6) Sample #5 after sintering in vacuum at 400 °C for 30 minutes.

5. Properties of activated carbon interacted with Si-organics

The results of XRD analyses are summarized in Figures 3 and 4. It is important to note that admixtures of different crystalline phases (calcite and unknown organic phase) and elemental amorphous sulfur (Fig.3, spectrum #1) in undoped activated carbon SKT-3S made interpretation essentially more difficult. Interaction with Si-organics at low temperature in air slightly changed the shape of XRD spectrum in low angle area (Fig.3, spectrum #2), however, the interpretation is unclear. Sintering in vacuum initiated chemical interaction between admixtures of sulfur and calcite and caused formation of CaS (Fig. 3, spectrum #3). Also, the destruction of unknown crystalline organic phase has been observed as well as quartz crystallization. (Fig. 3, spectrum #3).

Process of iodine doping initiated interaction between elemental sulfur and calcite that caused formation of anhydrite, CaSO_4 (Figure 4, spectrum #4). The chemical reason of this process so far is unclear. Interaction of iodine doped activated carbon with Si-organics at low temperature in air did not change essentially the XRD spectrum, excepts decrease of anhydrite reflections (Figure 4, spectrum #5). Further sintering of this sample in vacuum caused better crystallization of anhydrite (Figure 4, spectrum #6) and it was accompanied with complete loss of iodine. It is necessary to note that we used for this sample the low iodine doping level (57 mg/g) in order to provide the most reliable iodine sorption. The absence of quartz or Si-carbide phases in this sample means as assumed a significant evaporation of Si-organics before its chemical destruction or interaction with carbon. We might conclude, that activated carbon SKT-3S easily loses iodine at temperature which could provide some chemical interaction between Si-organics and carbon. There are still some uncertainties concerning the influence of chemical admixtures in SKT-3S to iodine loss.

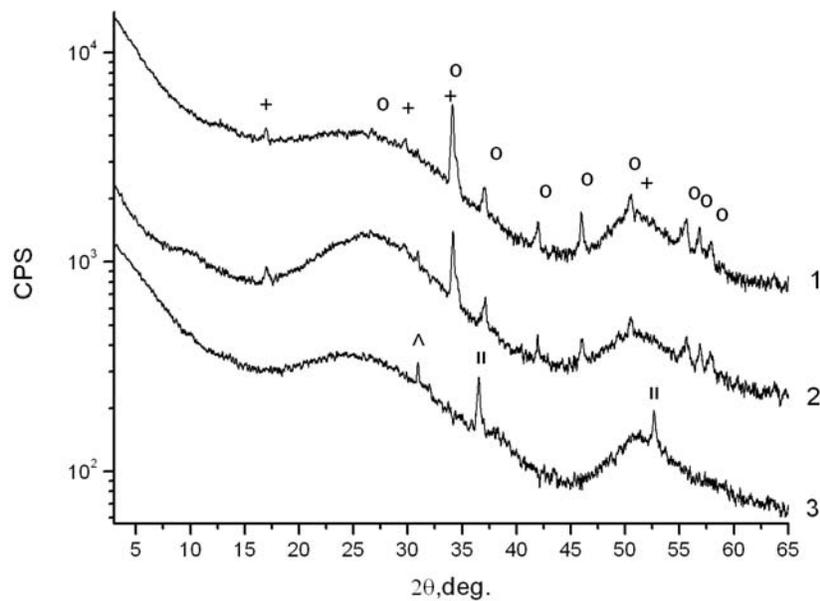


Figure 3. XRD patterns of **undoped activated carbon**: (1)- initial and (2-3) after interaction with Si-organics under different conditions. Peaks of calcite, CaCO_3 , are marked as “o”; unknown organic phase as “+”; calcium sulfide, CaS , - “II” and quartz - “^”.

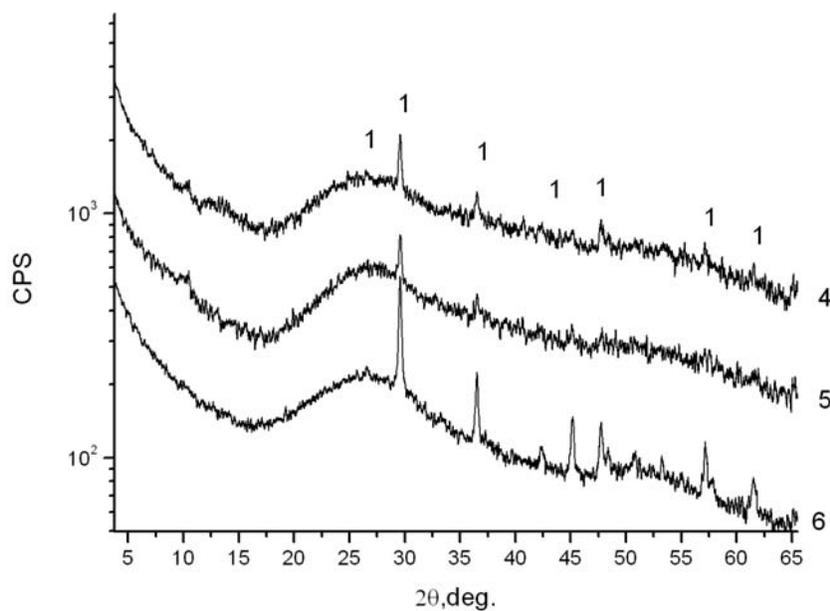


Figure 4. XRD patterns of **iodine doped activated carbon**: (4)- initial and (5-6) after interaction with Si-organics under different conditions. Peaks of anhydride, CaSO_4 , are marked as “1”.

6. Conclusions

The results obtained allow us to make the following conclusions:

- 1) Formation of crystalline phase Si_xC_y as a result of interaction between pure and iodine doped FCC and activated carbon SKT-3S has not been observed under applied conditions. Some evidences of possible formation of amorphous Si-C-phase were obtained only for FCC samples. It should be confirmed by further study using TEM method.
- 2) Sintering of pure and iodine doped FCC in vacuum before and after alteration with Si-organics caused essential (up to 50-60 %) increase of graphite content. However, no graphite formation was observed in pure and iodine doped activated carbon under similar conditions before and after alteration with Si-organics.
- 3) Study of new samples confirmed that iodine actively interacts with FCC. Doping of FCC with iodine causes destruction of phases based on benzene-like carbon rings such as graphite and fullerenes. Therefore, initial structure of FCC based on benzene-like carbon rings is transformed under iodine doping to the chaoite structure based on carbyne chains.
- 4) Laser beam affecting of pure and iodine doped FCC altered by Si-organics caused essential decrease of graphite content. Also, for undoped FCC the increase of fullerene content after laser impact has been observed.
- 5) Activated carbon SKT-3S easily loses iodine at temperature which could provide some chemical interaction between Si-organics and carbon.

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