



# Energeia



## The Role of F-400 Granular Activated Carbon in Scavenging Dissolved Copper Ions from Aqueous Solutions

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### INTRODUCTION

Adsorption of Copper from aqueous solutions using Granular Activated Carbon (GAC) F-400 loaded with Oxine and its derivatives was investigated. The results obtained from equilibrium studies showed significant uptake of copper. The equilibrium data for adsorption fitted well with the Freundlich Adsorption Isotherm. A comparative study of adsorption capacity of the various derivatives was also studied.

Metals of most concern in industrial wastes are Cd, Cu, Mo, Ni and Zn because of their tendency to accumulate in biological systems. In view of their high toxicity, persistence and increasing use, our group evaluated the effectiveness of the treatment process i.e., adsorption by Granular Activated Carbon, to remove copper ions in water. The reason for choosing this transition metal was that it forms one of the major constituents of industrial streams, due to easy detection procedures, and relatively less complicated behavior in an aqueous medium. Granular Activated Carbon F-400 gifted by M/S Calgon

## Carbon Surface Chemistry in Fly Ash Utilization and the Potential for Ash Beneficiation by Ozone

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One of the biggest challenges facing the fly ash utilization industry is the management of unburned carbon. Elevated carbon levels often accompany low-NOx retrofits of coal-fired power stations and can disqualify ash for its largest and most lucrative utilization market: as a pozzolanic additive in concrete. Thus by the "first law of unintended consequences," the NOx regulations designed to improve air quality have exacerbated another environmental problem — the need for land disposal of large volumes of unmarketable, high-carbon ash. Chronic problems with unburned carbon have prompted a significant research effort over the last several years on various aspects of the problem, including techniques for predicting residual carbon levels, material properties of residual carbon, post-combustion processes for carbon/ash separation or low-temperature burnout, and novel uses for high carbon ash or for concentrated residual carbon streams.

At Brown University we have been engaged for some years in an effort to understand the mechanism through which carbon in ash degrades concrete performance. In North America the most serious problem with carbon in concrete is its effect on the air entrainment process. Carbon adsorbs the surfactants or "air entraining admixtures" used to stabilize a system of fine air bubbles and thus impart improved freeze/thaw resistance in concrete. Our interest in the mechanism of this phenomenon began upon hearing field reports of local ash samples causing air entrainment problems despite low carbon levels (1-3%). In the past it was often assumed that low carbon level (or low LOI) was sufficient to ensure adequate air entrainment behavior, and this philosophy underlies current ash regulations, which are written in terms of LOI. After gathering a large set of ash samples from U.S. sites for laboratory testing, it became clear that there is significant variability in the specific surfactant absorptivity — i.e. the amount of concrete surfactant adsorbed under standard conditions *per gram of carbon*. Thus carbon content (LOI) alone is not always an adequate measure of how "good" or

## Carbon Surface Chemistry, (cont.)

“bad” an ash will behave in an air entrained concrete application, especially when considering a wide range of fuels (e.g. petroleum coke vs. low-rank coal) or in the presence of soot or fine particulate char. Our testing revealed that both carbon surface area and carbon particle size were key variables, the latter affecting the ease of access to the surface area lying in the particle interior regions.

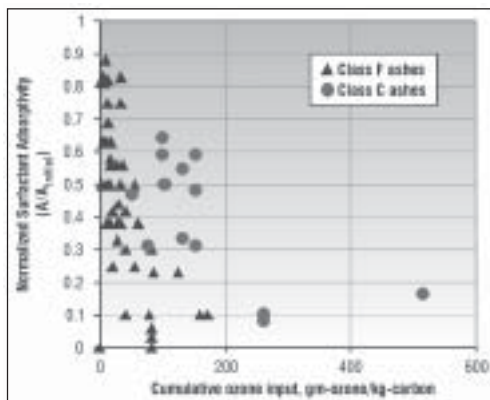
Some of our early data showed that air oxidation could reduce the affinity of unburned carbon for concrete surfactants, and that the effect is clearly visible at the point of incipient combustion — before any significant consumption of the carbon mass occurs! This data suggested that surface oxides on carbon reduce surfactant adsorptivity and thus improve ash quality. It is not surprising that surface chemistry should play some role, since the underlying phenomenon is adsorption of organic compounds from the aqueous phase — the same phenomenon underlying water treatment processes, where the presence of surface oxides on activated carbon is well known to influence adsorption behavior.

Recognizing the role of carbon surface chemistry in ash utilization opens the possibility of benefiting ash by intentional surface oxidation. Of the many possible oxidation methods, ozone has been the focus of our studies since it allows treatment of the ash in the dry state at near ambient temperatures, and the large-scale production of ozone is a proven commercial technology.

Figure 1 shows how ozone treatment reduces the surfactant adsorptivity that is the cause of air entrainment problems. This laboratory data was obtained by passing ozone-containing air (500 ppm - 2%) through small fixed beds of ash and measuring the adsorptivity by a simple titration procedure known in the ash industry as the “foam index test.” This simple ozone treatment is found to be effective for a wide variety of ash types and for several commercial air entraining admixtures and other surfactants.

## SURFACE CHARACTERIZATION

Ongoing work at Brown is focusing on the mechanism by which ozone passivates carbon surfaces. The question of mechanism here is not wholly academic — indeed it arose



*Figure 1. The effect of ozone treatment on normalized surfactant adsorptivity (foam index) of commercial fly ash samples. Class C ashes often require more ozone per gram of carbon due presumably to the higher specific surface area of class C unburned carbons (typical value 300 m<sup>2</sup>/g) relative to class F unburned carbons (typical value 50 m<sup>2</sup>/g).*

during the application phase for our US Patent (6136089) on fly ash ozonation, when the examiner presented the following counter argument (paraphrased): “since patents already exist for the beneficiation of ash by air oxidation (i.e. carbon burnout processes), it is obvious to those practiced in the art that ozone, an even stronger oxidant, would be capable of achieving the same effect.” However, ozone does not achieve its desired effect through carbon burnout — we typically see insignificant weight loss or even slight weight *gain* — but rather achieves its effect through modification of surface chemistry, a concept that is quite different and was

Sample	Atom-% O*	Atom-% C*	Surfactant Adsorpt, ml
Untreated carbon black	1	98	10
Air oxidized at 440 °C	7	92	3.5
Treated in 2 wt-% ozone	10	89	2

\* near-surface elemental compositions; balance sulfur

*Table 1. XPS results on raw and treated carbon black*

Sample	Surface Energy (mJ/m <sup>2</sup> )	Dispersive Component (mJ/m <sup>2</sup> )	Polar Component (mJ/m <sup>2</sup> )	Surfactant Adsorpt. (ml)
Untreated carbon black	21.8	20.9	0.9	10
Air oxidized at 440 °C	27.0	22.7	4.3	3.5
Treated in 2 wt-% ozone	32.4	24.4	8.1	2.5

\* determined by Owens-Wendt theory using contact angle measurements for benzyl alcohol and nitromethane as standard reference liquids

*Table 2. Surface energies\* of carbon black samples oxidized under similar conditions to fly ash carbon.*

ultimately deemed patent worthy. Some of our ongoing work uses carbon black as a model material that shows the same effect of ozonation, but is largely free from inorganic matter that interferes with modern surface characterization methods. The XPS results in Table 1 show greatly enhanced oxygen contents in the near-surface regions of carbon black samples ozonated under the same conditions used for fly ash carbon.

Surface energy analysis in Table 2 shows greatly enhanced polar contributions in carbon black samples ozonated under the same conditions used for fly ash carbon. Air oxidation at 440 °C is also seen to increase surface polarity and to decrease adsorptivity, but to a lesser extent than ozonation. The dispersive component of surface energy is observed to increase as well, though only slightly, so the net effect of oxidation is a rather large increase in total surface energy (polar plus dispersive).

Finally, through measurement of vapor adsorption isotherms, we find that ozonation decreases the total surface area for some ash samples, but not by a large enough factor to explain the observed suppression of surfactant adsorptivity. For carbon black, ozonation does not decrease surface area, but it is still quite effective at reducing surfactant adsorptivity.

## MECHANISM OF SURFACTANT ADSORPTION AND ITS SUPPRESSION BY OZONE

For aqueous phase adsorption phenomena in general, there are several ways that surface oxidation may suppress adsorption:

1. “micropore blockage” – surface oxide formation decreases total area by blocking fine pores or pore mouths.
2. “electron withdrawal” – the addition of electronegative oxygen atoms to graphene layer edges with draws electron density from the aromatic systems and reduces dispersion forces that bind the surfactant to the surface.
3. “electrostatics” – the acidic nature of most carbon surface oxides leads to a negatively charged surface in the high-pH concrete solution. The net negative surface charge repels the AEA molecules, which are anionic (negatively charged) surfactants.

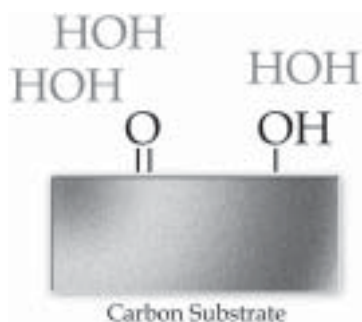
## Carbon Surface Chemistry, (cont.)

4. “reduction of hydrophobic forces” – introduction of oxides destroys non-polar surface area that is responsible for adsorption, leaving only polar surfaces that are hydrophilic and solvated (hydrogen bonded to water) and thus effectively unavailable for surfactant adsorption.

The recent work allows us to evaluate these competing explanations for our system. Mechanism 1 (pore blockage) is a likely contributor for at least some fly ash samples, but is not believed to be the primary mechanism, since carbon black exhibits the same beneficial effect of ozonation, but does not show any decrease in area. The different area behavior of carbon black and fly ash carbon is not surprising, since most area in carbon black is mesoporous, lying on the external surfaces of the nanoscale primary spheres, thus giving little opportunity for micropore blockage by surface oxides.

Mechanism 2 (electron withdrawal) can be ruled out by the surface energy results derived from wetting studies (Table 2). Ozonation is seen to add a polar component to the surface energy without decreasing the dispersive component. Indeed the dispersive component increases slightly and the overall effect is a large increase in total surface energy. Thus we expect ozonation to slightly *enhance* the dispersive attractive forces for adsorption, not suppress them.

Mechanism 3 (electrostatics) may contribute for anionic AEAs, but is not



*Figure 2. Sketch of the hydrophilic carbon surface following ozone treatment showing solvated surface sites no longer available for surfactant adsorption. Since hydrophobic forces alone are sufficient for surfactant adsorption, we believe that the key to passivation is the destruction of the active hydrophobic (non-polar) surface and its subsequent solvation.*

likely the prime driver since we observe the same effect of ozone when using the non-ionic surfactant Tergitol. This uncharged molecule should not be greatly affected by changes in net surface charge.

Mechanism 4 (reduction of hydrophobic forces) is believed to be the primary mechanism, in part by the process of elimination and in part from basic considerations about the nature of surfactants. Unlike other soluble organics, surfactants have a highly insoluble nonpolar part (“tail”), which is strongly driven by hydrophobic forces to leave the aqueous phase. It thus collects at the air interface, chiefly in bubble cavities, and on any other available non-polar surface. Because of its very low solubility we believe the surfactant is not particularly selective about the nature of the non-polar surface and does not require strong attractive adsorption interactions. In the case of the air interface the surfactant molecules collect by these “hydrophobic forces” alone with no attractive forces whatsoever between the surfactant and the interface. We therefore believe that the adsorptivity of carbon is directly related to the fraction of its surface that is hydrophobic (non-polar) with other characteristics of the surface being secondary. Ozonation destroys this non-polar surface and replaces it with an oxidic surface that is hydrophilic and capable of strong hydrogen bonding with the solvent (Figure 2). Since adsorption from solution is intrinsically a competitive process in which the surfactant and solvent (water) compete for sites, the water molecules have a strong advantage over the surfactant molecules on oxide-covered surfaces and the overall effect is suppression of the surfactant adsorptivity.

### PROSPECTS FOR A COMMERCIAL ASH OZONATION PROCESS

In parallel with the fundamental studies described above, efforts are underway to develop an ash beneficiation process based on this principle. The Electric Power Research Institute (EPRI) has assembled a multi-organizational team consisting of PCI-Wedeco (West Caldwell, NJ), the world’s leading manufacturer of large-scale ozone generating systems, Fuller Bulk Handling Corporation, manufacture of equipment for solids blending, conveying, and storage, Brown University with expertise in process chemistry, and

the utility companies PPL Generation and Dairyland Power with problem ash streams and access to potential markets for beneficiated product. A key issue in the development is the proper choice of a gas/solid contacting method, and the team is currently evaluating conveying systems, blenders, and fluidized beds.

For some in the ash industry, ozone may seem like an exotic laboratory reagent, but modern large-scale ozone processes are now commonplace in wastewater treatment, bleaching operations, food disinfection, and other niche applications. Many of these processes involve on-site workers. Ozone, while toxic, is easily managed and the industry has an outstanding safety record. There is plenty of competition in the ash beneficiation arena, with a variety of commercial or developmental processes based on triboelectrostatic carbon removal, carbon burnout in fluidized beds, and froth flotation. The most likely role for ozonation is to occupy a market niche for ash streams with marginally high carbon (3-5%) or for those with low carbon levels (1-2%) but where high specific carbon activity still leads to air entrainment problems — a situation that has been documented at a number of utility sites. Ozonation is particularly suited for these ash streams since they pose challenges for the competing processes — low-carbon ashes are hard to fractionate at high yield using electrostatics, and they require supplemental fuel in burnout processes. Additionally they are often below the regulatory threshold for carbon content (LOI), so the main disadvantage of ozone — namely that it leaves the unburned carbon in place — does not pose a regulatory hurdle.

### Acknowledgments

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## The Role of F-400 Granular Activated Carbon (cont.)

Carbon Type	Surface Area N <sub>2</sub> -BETm <sup>2</sup> /g	Particle Density g/cm <sup>3</sup>	Apparent Density g/cm <sup>3</sup>	True Density g/cm <sup>3</sup>	Pore Volume cm <sup>3</sup> /g	Porosity
F-400	998	0.7950	0.4800	2.308	0.825	0.65

Table 1. Properties of GAC F - 400

Corporation, Pittsburgh, USA (Table 1) was chosen for this work because it is a widely used GAC and has recognized applications for metal recovery and wastewater treatment. Choosing Oxine and its derivatives was primarily based on the fact that it is a well-known chelating ligand whose chemistry of chelation is well understood. Secondly the ligands used are easily synthesized or commercially available and readily form aqueous solutions. Attempts have been made to increase the selectivity and sensitivity of these reactions by using derivatives.

An exhaustive literature survey provided only few cases in which ligand-loaded carbon has been used to scavenge metal ions. Vanderborght and Grieken used 8-hydroxy quinoline on complex trace elements, which were then adsorbed on activated carbon. Natarajan separated Cu ions in an aqueous medium either alone or in admixtures with other divalent ions using GAC containing adsorbed 8-hydroxy quinoline-5-sulphonic acid. Attempts also were made to improve scavenging metal ions from aqueous solutions using ligand-adsorbed carbon beyond adsorption normally encountered with commercial carbons alone.

### Experimental

Standard solutions of the basic oxines (0.0015 M) and the sulphonated oxines (0.002 M) were prepared. A standard Beer's law plot was established by measuring absorbance of dilute solutions at 350 nm. For the metal ion solution an approximately 0.002 M solution of BDH grade cupric chloride solution was prepared. It was standardized against an alkali solution using pH titration. A calibration plot was established spectrophotometrically using standard methods.

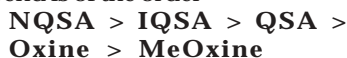
The adsorption experiments for the ligands were carried out in the batch mode using bottles of 250 ml capacity with 200 ml of ligand solution and the GAC weight varied from 0.5-1.5 g respectively. The bottles were shaken for a predetermined period of 7-8 hours or a maximum of overnight for some systems at 25+/-1 °C in a mechanical

shaker. The solutions were filtered and the final concentration of ligand was determined spectrophotometrically to ensure uniformity of ligand adsorbed for subsequent experiments with the copper metal ion.

The ligand-loaded GAC's ability to scavenge Cu ions from aqueous solutions was then studied. The ligands adsorbed on the carbon surface practically behave like a solid phase capable of scavenging Cu ions from solution. To the ligand-loaded carbon was added 100 ml of Cu solution whose concentration ranged between 25 mg - 120 mg /l. The period of agitated contact was eight hours. For all experiments initial and final concentrations of Cu was determined spectrophotometrically using the Na - DDC method. Since carbon is not expected to function effectively as a basic scavenging material for the Cu in solution, it was proper to substitute the weight of carbon by the millimoles of ligand present on the carbon while calculating the copper scavenged.

### Results and Discussion

Table 2 gives an idea of maximum values (saturation values) of  $q_e$  as obtained in the work. These values of  $q_e$  represent the maximum amount of Cu that a millimole of the ligand can hold in the carbon. Under the present circumstances it is observed that the trend is of the order



showing that MeOxine can remove only a small amount of Cu from solution compared to Oxine. This could be due to adsorption of MeOxine on the surface of carbon through the

S. No	Ligand	$q_e$ mg/millimole
1.	Oxine	18.0
2.	MeOxine	13.0
3.	QSA	26.0
4.	IQSA	31.0
5.	NQSA	37.0

Table 2. Amount of Cu held/ millimole of ligand on F - 400 GAC.

methyl group, which would mean that the ligand's hydroxyl and nitrogen chelating centers would be oriented facing the surface of carbon and thus would not be easily available to form a 1:2 complex. On the contrary Oxine and its other derivatives are probably flatly oriented on the surface of GAC, providing proper orientation of the ligand molecules to facilitate formation of a 1:2 complex with the copper on the surface of the GAC. As far as the sulphonated ligands are concerned, they all form strong complexes with copper. The trend in the  $q_e$  values can be attributed to the bulky nature of iodo and nitro derivatives. QSA, being smallest, is able to penetrate deep into the pores of GAC, making it less available through proper orientation for forming a 1:2 complex with Cu ions on the surface of GAC, thus showing less  $q_e$  value. On the other hand the bulkier iodo and nitro derivatives are unable to penetrate the pores and remain on the surface of GAC, thereby making it easier to form the 1:2 complex with copper. This is in agreement with the  $q_e$  values experimentally determined. This work shows that the larger ligand molecules are restricted in the wider pores at the mouth and even though NQSA is adsorbed to a marginally larger extent than IQSA on the GAC, it can remove more copper than QSA probably because of proper orientation of the ligand molecules in the adsorbed state.

The results obtained from equilibrium studies showed that there was significant uptake of copper metal ions by ligand loaded F-400 GAC. Thus a conclusion can be drawn that the pore structure of the GAC and chemistry of the molecule probably contribute much to the common metal pollutant removal, such as copper, through ligands adsorbed on the surface of GAC from aqueous solutions.

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## Integrating Research and Education through

# mentoring

—advice from one who has done it—

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Southern University and  
A&M College, Baton Rouge and  
Director of “The Timbuktu Academy”*

From the standpoint of a student led through a discovery process, there is little difference between research and discovery learning (or teaching). The difference, if any, stems from the fact that, in classroom activities, the teacher knows where we are heading! For the last 10 years, significant changes have occurred in K-12 education. The various educational reforms that are somewhat synthesized in the National Science Education Standards (NSES) have several features in common:

- ▶ They emphasize teaching and learning processes that *engage* the learners as much as possible;
- ▶ This emphasis partly rests on the fact that the behaviorist and cognitivist approaches to learning, far from being in contradiction, are complementary facets of the same reality;
- ▶ These reforms therefore support the following principles:
  - (a) Emphasis on engaging the learner to develop understanding and operational knowledge as opposed to almost exclusively utilizing “lectures,” rote memorization, and the related recall of a plethora of disjointed propositions of declarative knowledge;
  - (b) Emphasis on thematic approaches in curriculum design and delivery and on conceptual understanding as gauged by outcome assessment methods; and
  - (c) High expectations for all students.



A corollary of the above emphasis on processes is that mentoring, including research participation, is integral to learning. No amount of talking about complex processes can substitute for actual hands-on inquiry. This fact is apparent in graduate education where the coupling between research and education is obvious. The research advisor/advisee relationship is very analogous to apprenticeship, a form of mentoring. When cultivated properly, it is an intrinsic mentoring relationship. [In fact, it should be noted that, even for faculty members, to conduct research is to be engaged in continuing education!] The good news is that the aforementioned pertinence of research to graduate education also holds at the undergraduate level, inasmuch as undergraduate students are expected to acquire not only some knowledge but also some habits of mind that are inherent to research or to practicing science.

Several national and state efforts are currently aimed at enabling the integration of research and education for undergraduate students in science, engineering, and mathematics (SEM). In particular, the Alliances for Minority Participation (AMPs), funded by the National Science Foundation (NSF), endeavor to engage minority SEM students in research as a means to not only retain them but also to enhance the likelihood of their pursuing graduate degrees. These students are often immersed in a systemic mentoring environment that ensures their academic and social integration. The 10-step or 10-strand systemic mentoring model of the Timbuktu Academy and of the Louisiana AMP are available on the web ([www.phys.subr.edu/timbuktu.htm](http://www.phys.subr.edu/timbuktu.htm) and [www.ls-lamp.org](http://www.ls-lamp.org)). A research lab, in the above sense, is a classroom for a “community of learners” that typically includes faculty, post-doctoral fellows, teachers, graduate students, and undergraduate students. A failure to grasp the above view often leads to the perception that research and education are not only different but also in conflict! With the correct understanding and with mentoring, they complement and enable one another!

The first document on the Timbuktu Academy web site describes its objectives, design principles, paradigm, programs, activities, and results. Established in 1990, it is named after the former University of Timbuktu, a bastion of scholarship in the middle of the second millennium, in the city of Timbuktu, on the banks of the majestic Niger River, in Mali, West Africa. Southern University and A&M College in Baton Rouge (SUBR) is similarly located on the banks of the mighty Mississippi! Every summer, the Academy engages 120 to 200 elementary to high school students in standard-based academic enrichment activities in English, mathematics, science, and related subjects. An introduction to research is always a part of the activities. The Timbuktu Academy engages 50 to 100 undergraduate SEM students in its systemic mentoring activities every year. Given our view of research as a necessary component of learning SEM, these

## Integrating Research and Education, (cont.)

students are involved in research during the academic year or the summer. Every summer, approximately 45 of these students are placed in premiere federal, industrial, and university research labs around the country. Two were at the University of Kentucky in 1999 and in 2000.

The Louisiana AMP, to a great extent, replicates the above model of the Timbuktu Academy at 11 colleges and universities, including the Research I Universities.

We urge the reader to consult the referenced web site in order to appreciate the fact that we placed mentoring on a rigorous, scientific basis as opposed to an anecdotal or incidental approach. In particular, our publications [Education, Vol. 115,

No.1, page 31-39, 1994; and College Teaching, Winter 2000] established a paradigm for systemic mentoring and one for problem solving. The 'power law of performance of cognitive science' plays a pivotal role in these paradigms. It is the basis of high expectations for all students. The above scientific approach means that the Academy's success is gauged mainly by external standards (i.e., ACT/SAT, GRE test scores; presentations; refereed publications). The web site of Louisiana AMP provides a road map for mentoring by individual faculty members and for establishing systemic mentoring in departments and research laboratories. Mentoring is systemic if it is woven into the core activities of an organization or department. Given that "we do what is checked, valued, and rewarded," the

establishment of systemic mentoring often requires that the group leader, the laboratory or department head, and the other individual with decision and budget authority set the proper expectations verbally and in writing. Positive incentives (funds, awards, recognition, etc.) over the years will modify behaviors in the systemic mentoring direction! With the advent of electronic universities, this systemic mentoring may well be the greatest competitive edge of many regular educational and research institutions—as far as recruiting undergraduate and graduate student is concerned!

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