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## LOW-TEMPERATURE PLASMA TECHNOLOGY AS PART OF A CLOSED-LOOP RESOURCE MANAGEMENT SYSTEM

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### ABSTRACT

More comprehensive resource management systems must be developed if the United States is to continue extraterrestrial exploration involving extended lengths of time in space. Testing was performed to obtain additional information concerning the feasibility of using a low-temperature plasma reactor as the centerpiece of a closed-loop processing (CLP) resource management system. Low- and high-ash carbon, freeze-dried human feces, sunflower, and plastic were processed in a low-temperature oxygen plasma reactor system in both stationary and agitated modes. Conversion of carbon, hydrogen, and nitrogen to gaseous species was determined for each of the test materials. Sample agitation greatly reduced the residence times required for species conversion. Virtually complete carbon conversion was achieved at residence times ranging from 2 to 8 hr. The inorganic matrix was unchanged by the processing technique. Based upon the results of this testing, scale-up and further testing of this technology are warranted.

### INTRODUCTION

More comprehensive resource management systems must be developed if the United States is to continue extraterrestrial exploration involving extended lengths of time in space. Three types of resource management systems are available for this type of mission: 1) systems in which consumables such as oxygen and food are not recycled, 2) totally closed-loop systems in which all consumables are recovered and reused in some manner, and 3) a combination of 1 and 2. The quantity of consumable material to be recycled (i.e., 50% or 90%) and/or ferried back and forth from Earth will be based primarily on the mass, volume, and energy requirements of the closed-loop processing (CLP) system. An extraterrestrial base will require a CLP system capable of integrating all functions necessary to support life in a remote setting, including manufacturing and biological by-product processing.

A low-temperature plasma reactor is capable of oxidizing or reducing the organic components of a stream while leaving the inorganic matrix unaffected. In effect, separation and conversion take place in a single reactor. The organic fraction of plants, human waste, and plastics can be converted to gases, while the inorganic fraction remains virtually unchanged. The inorganic materials can then be recycled directly to other operations or subsystems.

The feasibility of using this technology as part of a CLP resource management system can be measured by reactor size, efficiency, and energy requirements, as well as by the capability of the reactor to be closely

integrated into the closed-loop system. Stationary-bed batch tests were performed previously at EERC to determine conversion rates and efficiencies for a variety of organic wastes (ref. 1). The results of these tests showed that the technology held promise as part of a CLP resource management system, with its ultimate potential dependent upon lowering residence time requirements. The current research focused on determining if the residence times could be reduced to a point where it is likely that scale-up and reactor system optimization could result in a small, energy-efficient system capable of being closely integrated into a closed-loop resource management system.

## METHOD AND MATERIALS

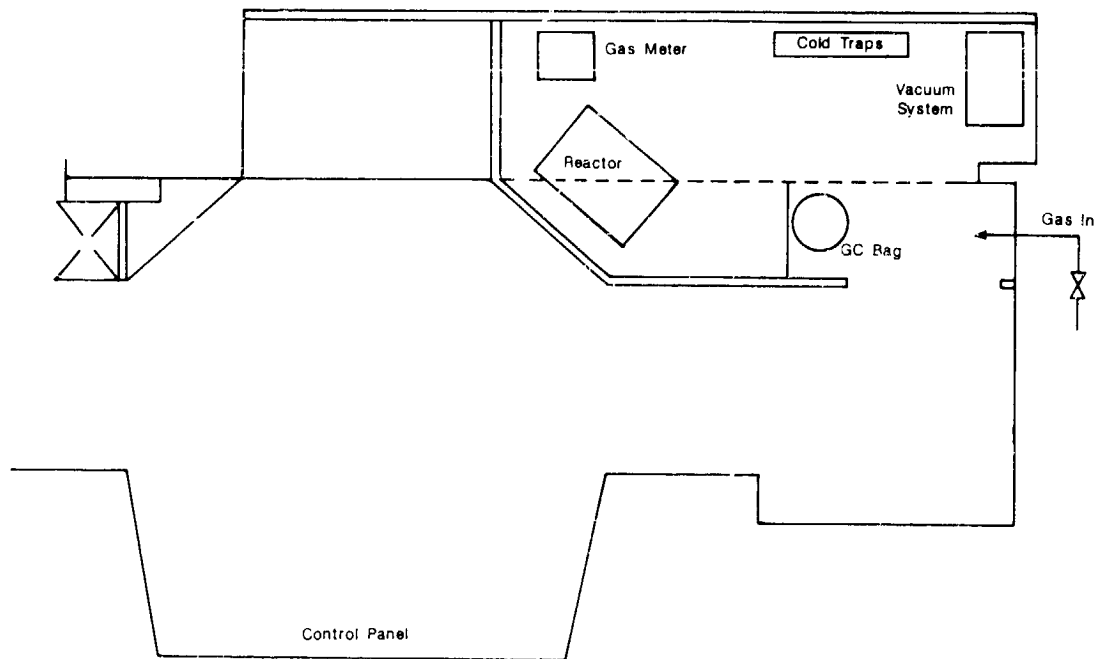
### Equipment

The low-temperature plasma tests were performed using three pieces of equipment: a radio frequency (rf) generator, a reactor center, and a vacuum system. The rf generator and the reactor center were manufactured by the International Plasma Corporation. The rf generator can generate up to 500 W of rf power at a frequency of 13.6 MHz. The reactor center houses the reactor chamber, the electrodes that generate the plasma field, and a control panel. The floor plan of the work area supporting this system is shown in Figure 1. The control panel is used to regulate the gas flow and the vacuum in the reactor chamber. It also balances the load on the rf generator. The quartz reactor chamber is eight inches long and four inches in diameter and has been modified to contain the hardware necessary for agitation during operation. A vacuum pulls the plasma-producing gas through the chamber, while the rf energy is transmitted across the electrodes surrounding the sides of the reactor chamber. The reactor chamber is shown in Figure 2.

As part of earlier research, the reactor chamber was fitted with three types of agitation. The first was a fritted quartz cylinder attached to a rotary coupling (the reactor agitation system originally proposed for this testing). Samples were placed inside this cylinder and tumbled during processing. Two other methods of sample agitation during processing were also established with the existing reactor system.

The second device consisted of a pair of rotating aluminum trays. The trays were each constructed with a lip on one longitudinal edge and were aligned as shown in Figure 3. They were attached to the rotary coupling and fitted inside the quartz reactor chamber. At this small scale, some of the particle sizes of the samples tested were not large enough to overcome the electrostatic forces which held them to the aluminum plates. To overcome this problem, the system was "internally" agitated. The method chosen was essentially a ball mill. An inert solid (sand or silica gel) was added to the test material to provide a mixing, crushing action. It also ensured that some type of sample would be present at extinction for analysis.

The third tumbling device consisted of a thin blade of aluminum which was attached to the rotary coupling. The blade continuously scraped the reactor chamber walls, thereby agitating the sample, as shown in Figure 4. This method also made use of the addition of an inert solid to the test materials for the purpose of providing further mixing action.



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Figure 1. (a) Floor plan of low-temperature plasma system work area.  
(b) Low-temperature plasma system control area.

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Figure 2. Low-temperature plasma system quartz reactor chamber.

During the tests, nominal reactor chamber pressure was held at 67 to 107 N/m<sup>2</sup>, rf power was set at 100 W, and the gas flow rate was less than 0.1 cc/min.

#### Experimental Method

Irrespective of the agitation method used, sand or silica gel was added to the test material in a mass ratio of 9:1. The composite sample was then loaded into the reactor. When the aluminum trays were used, the sample was placed on the bottom tray. As the trays rotated, the sample was dumped from one tray to another, falling through the plasma as it did so. At the completion of the test, the trays were removed and the sample collected. Any sample that had fallen off of the trays was reclaimed from the bottom of the reactor chamber and added back to the product container.

In the case of the method involving the rotating aluminum blade, the sample was loaded onto, and collected from, the bottom of the reactor chamber. This method was used for the majority of the kinetics tests involving agitation.

To provide a yardstick against which to measure the results of the tests involving agitation, tests were also performed in a stationary mode. The same quartz reactor chamber was used for these tests, but without agitation. Instead, the sample was loaded onto quartz trays which were placed inside the reactor chamber.

Quality control measures taken during this study are described in reference 2.

feed). These test points were not included in the plots. As expected, conversion to gaseous products generally took place more rapidly when samples were agitated, although the difference between the results of the agitated and stationary tests was more pronounced for some of the test materials.

Figure 5 shows the conversion of carbon in low-ash carbon as a function of time. In this instance, there was a rather dramatic difference between the results of the stationary and agitated tests. This can be compared with the conversion of carbon in high-ash carbon as a function of time, shown in Figure 6. The same trend is noted in that higher conversion levels were attained for the high-ash carbon sample when it was processed in the agitated reactor configuration. However, under stationary conditions, carbon conversions were higher for the high-ash carbon than for the low-ash carbon.

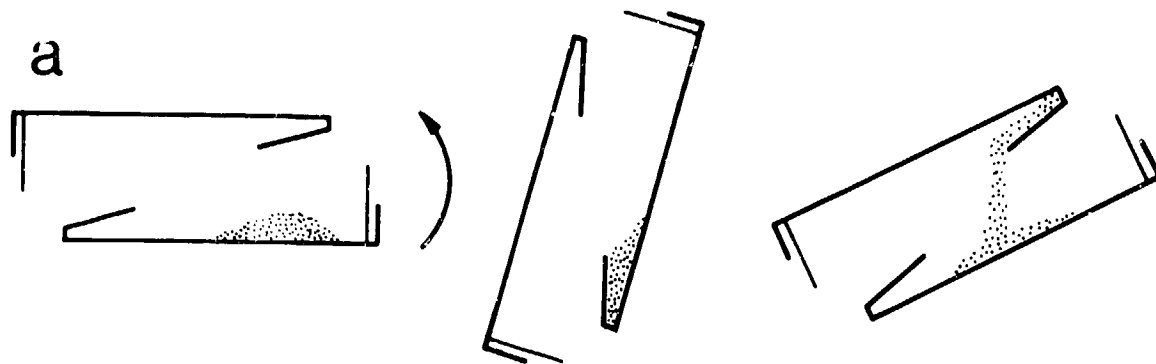
Of the five test materials, it was more difficult to obtain a representative sample of freeze-dried human fecal material for analysis due to the variations in particle size within the composite sample. As such, only one viable test point was obtained for the agitated tests. Conclusions can still be drawn from the plot shown in Figure 7. From the fecal material data point, it appears that the carbon conversion rate was enhanced by agitation and was of the same order of magnitude as that observed with carbon.

The results of processing the sunflower samples are presented in Figure 8. Again, agitation enhanced the conversion of carbon to gaseous species. The carbon conversions obtained for the sunflower samples were higher than those of any other materials tested within the one-hour kinetics evaluation series.

Conversions of hydrogen and nitrogen to gaseous products were also determined for the sunflower samples and are shown in Figures 9 and 10, respectively. As was the case with the carbon, agitation hastened the conversion of both the hydrogen and the nitrogen. Figure 10 indicates that the nitrogen conversion rate was more rapid during the first 20 min of agitated processing.

The plastic processed consisted of ground plexiglas. During the agitated processing, the particles had a tendency to melt together, making it difficult to contact the sample with the plasma. However, one viable test point was obtained for the agitated processing (60 min). As Figure 11 shows, this value is less than the 57% conversion obtained during stationary processing for 60 min. This is most likely due to the fact that the sample did not agglomerate during the stationary processing.

Samples of each of the test materials were processed to extinction using the rotating aluminum plate reactor configuration. Due to the tendency of the silica gel to stick to the reactor plates as a result of static electricity, the processing was performed using sand as the inert matrix. The product samples could not be analyzed for carbon, hydrogen, and nitrogen content because not enough test material remained. The time necessary for complete extinction varied from sample to sample, with the plastic requiring approximately 8 hr and the remaining samples requiring 3 hr or less. The five test materials are shown before and after processing in Figures 12 through 16.



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c

Figure 3. (a) Diagram of rotating tray agitation system.  
(b) Rotary coupling.  
(c) Rotating tray agitation system.

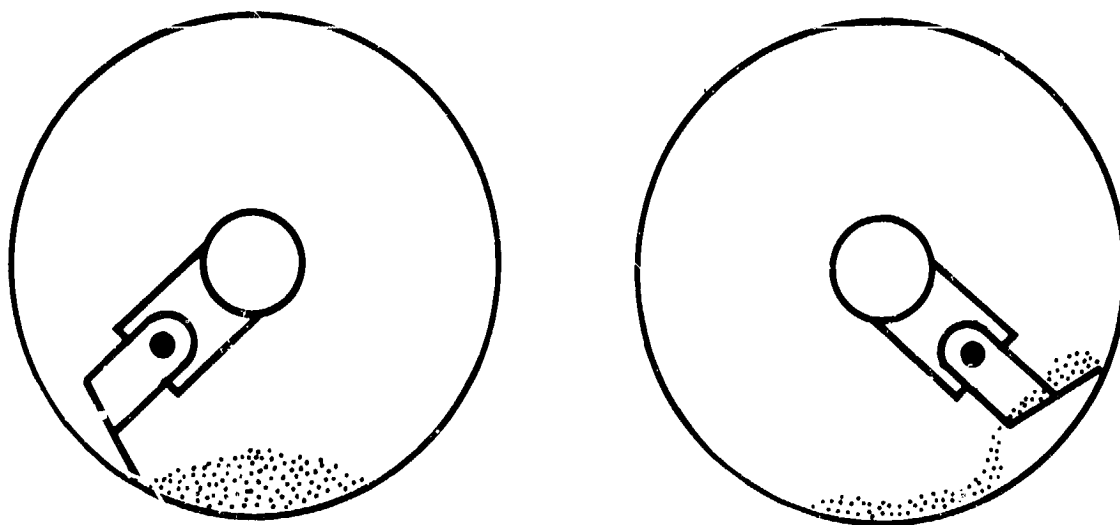


Figure 4. Diagram of aluminum blade agitation system.

#### Test Matrix

The tests were performed using oxygen plasma and the following materials:

1. Low-ash carbon
2. High-ash carbon
3. Freeze-dried human fecal material
4. Sunflower roots, stalk, and head
5. Plastic (ground plexiglas)

Tests were performed in both stationary and agitated modes for times of 20, 40, 60 min, and to extinction (less than 3 hr in most cases). The rotating aluminum blade method of agitation was used for the testing, except for the extinction tests, which were performed using the aluminum plate reactor. When possible, the product samples were analyzed for carbon, hydrogen, and nitrogen content.

#### ANALYTICAL RESULTS AND INTERPRETATION

Carbon, hydrogen, and nitrogen (CHN) contents were determined for the five feed samples and 29 of the products. The theoretical limits of calculations involving these results are the same as the detectability limits of the analytical instrumentation (i.e.,  $\pm 0.5\%$  for carbon and  $\pm 0.1\%$  for hydrogen and nitrogen).

Conversions of the carbon, hydrogen, and nitrogen to gaseous species were determined using the CHN information. The conversion data were plotted as functions of residence time for ease of interpretation. In some cases, negative conversions were calculated (i.e., the analytical results indicated that more carbon was present in the product than had been present in the

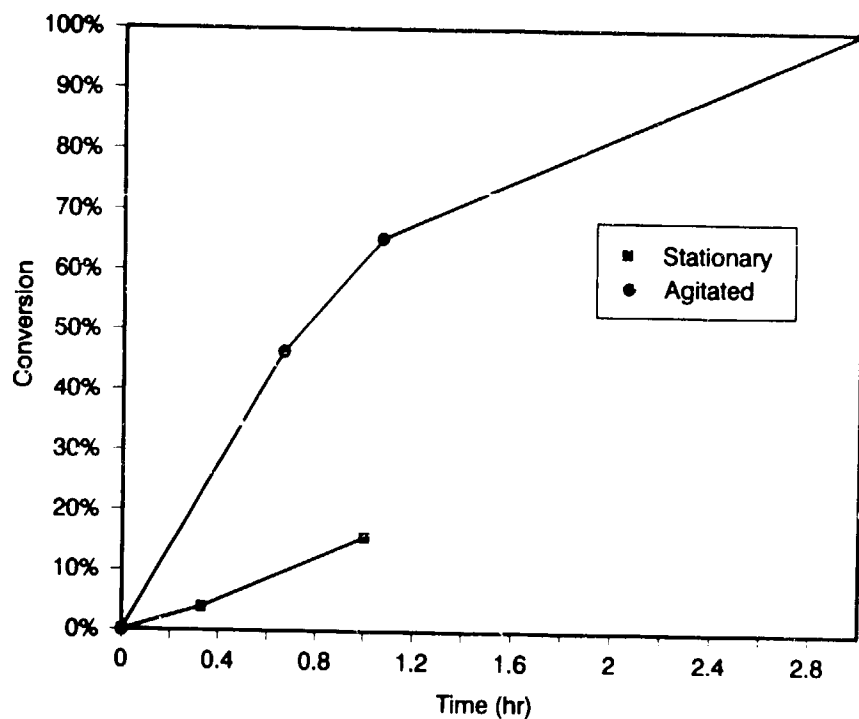


Figure 5. Carbon conversion in low-ash carbon sample as a function of time.

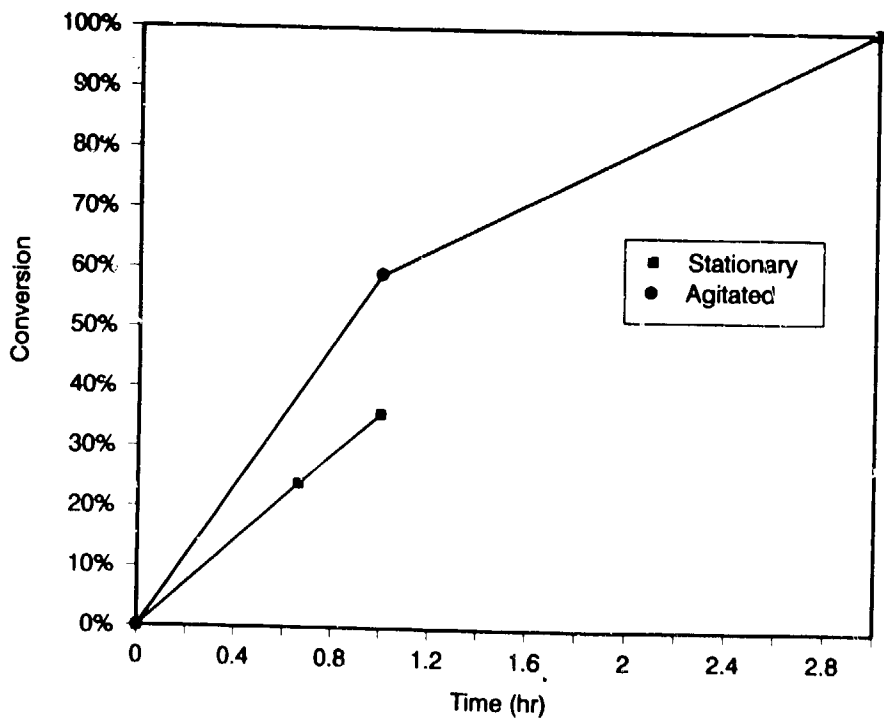


Figure 6. Carbon conversion in high-ash carbon sample as a function of time.



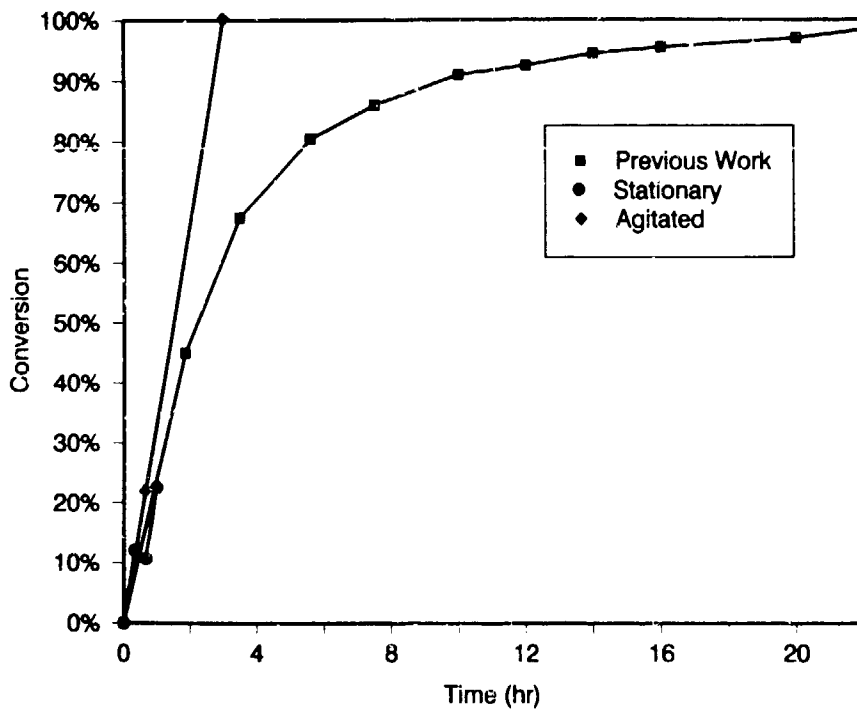


Figure 7. Carbon conversion in feces sample as a function of time.

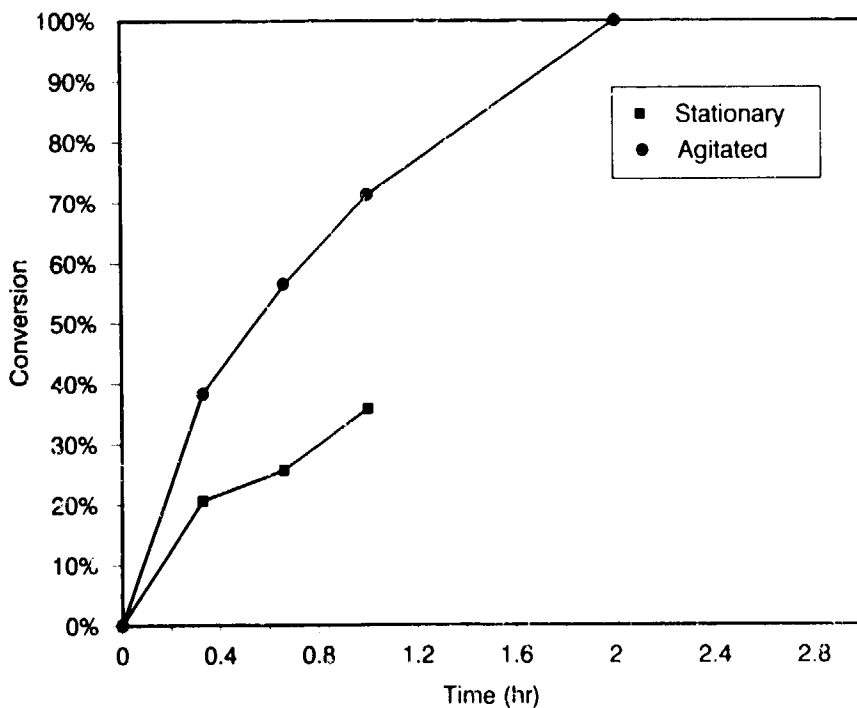


Figure 8. Carbon conversion in sunflower sample as a function of time.

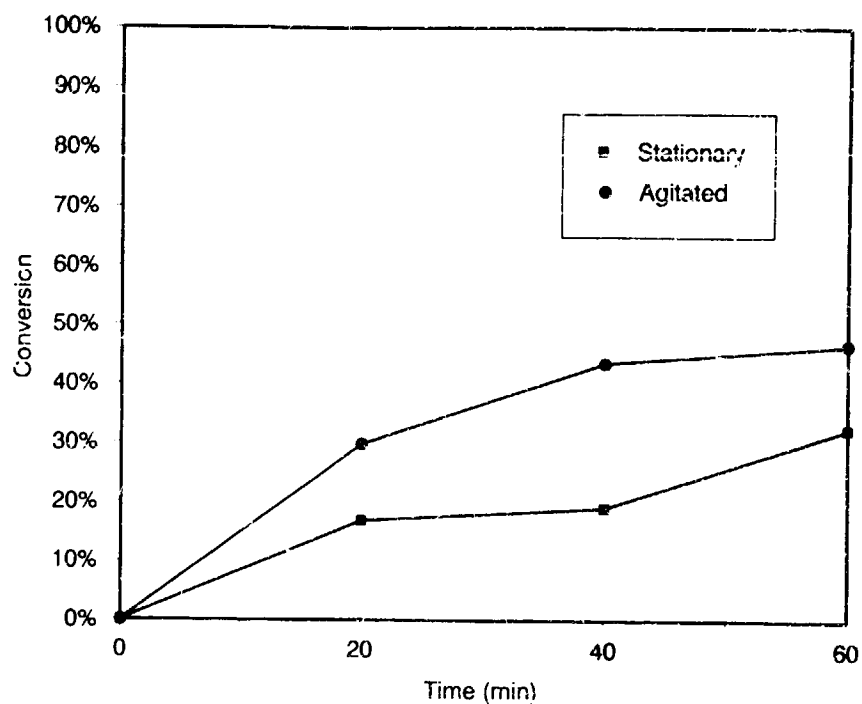


Figure 9. Hydrogen conversion in sunflower sample as a function of time.

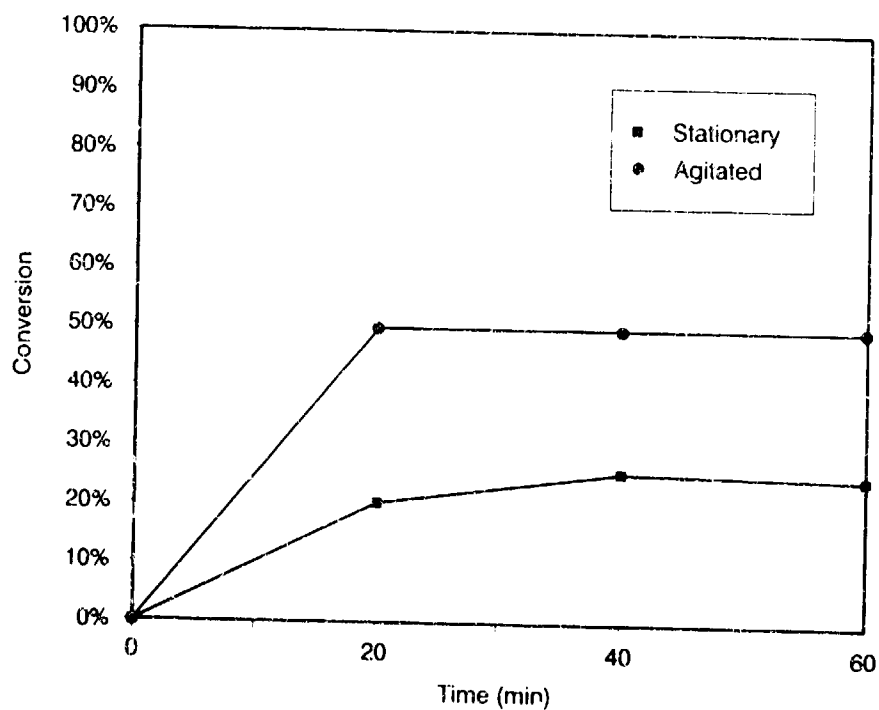


Figure 10. Nitrogen conversion in sunflower sample as a function of time.

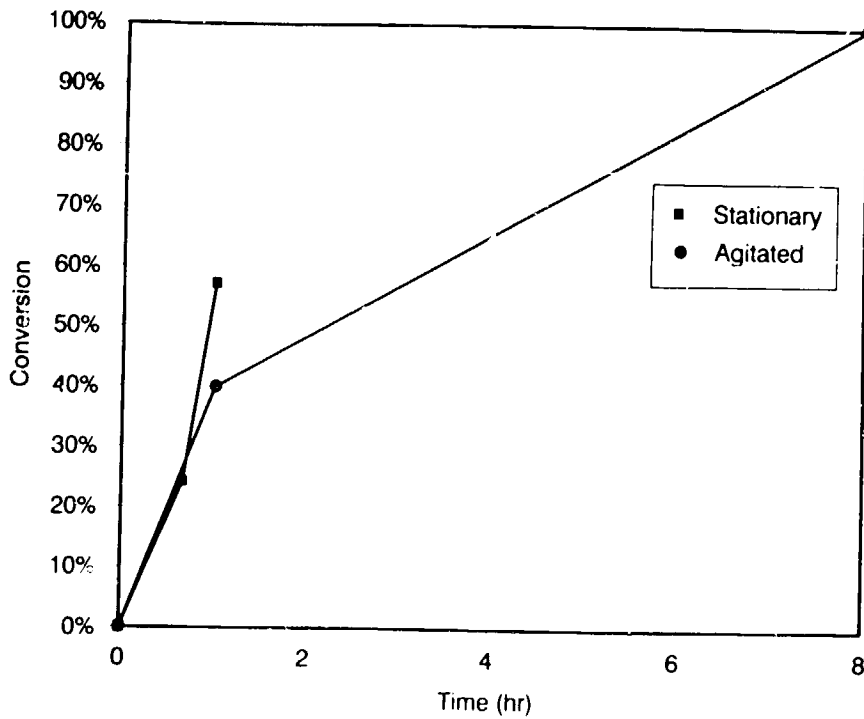


Figure 11. Carbon conversion in plastic sample as a function of time.

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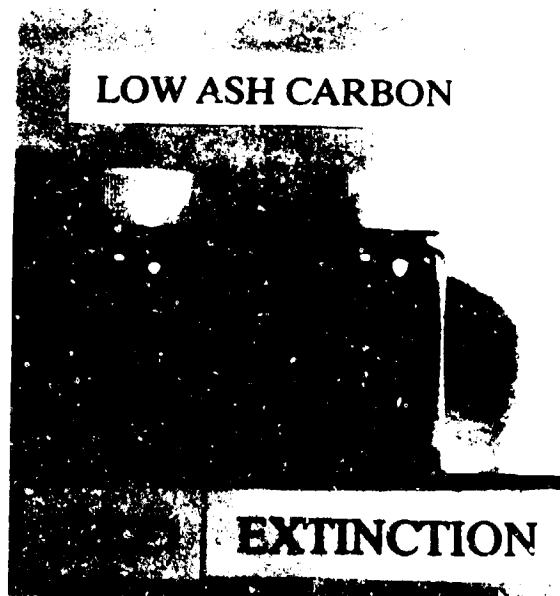


Figure 12. Low-ash carbon sample before and after processing to extinction.

HIGH ASH CARBON

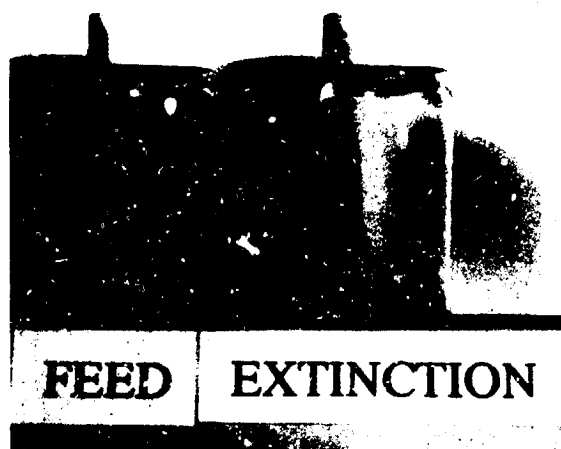


Figure 13. High-ash carbon sample before and after processing to extinction.

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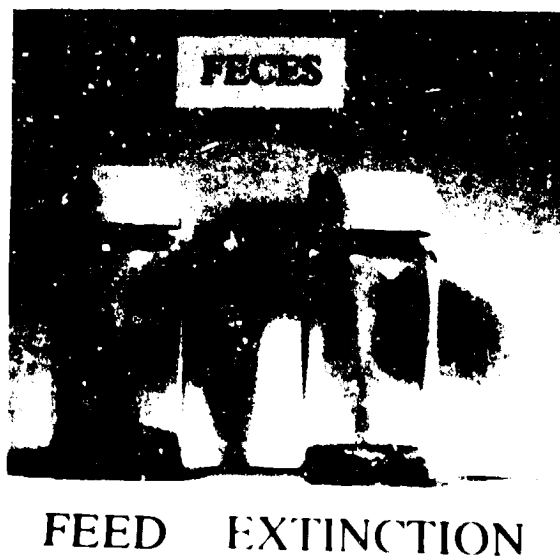


Figure 14. Freeze-dried feces sample before and after processing to extinction.

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SUNFLOWER

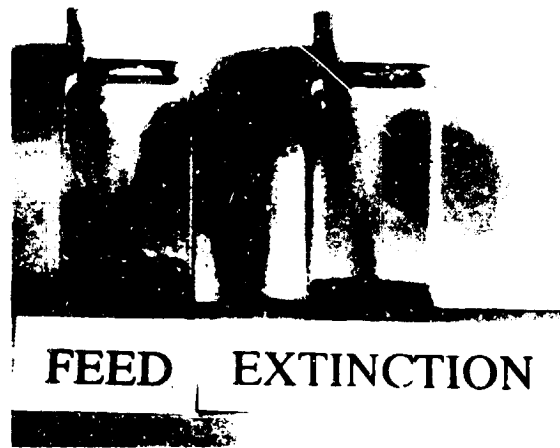


Figure 15. Sunflower sample before and after processing to extinction.



Figure 16. Plastic sample before and after processing to extinction.

The results obtained during this research were compared to those obtained during earlier bench-scale testing at EERC (ref. 1). The comparison is summarized in Table i. The products of the agitated runs were very similar in conversion and solubility characteristics to the products of the stationary tests performed during earlier work. The ranges of residence times required for complete carbon conversion are indicated in Figure 17. As the figure shows, the residence times required to completely convert the carbon were reduced dramatically by agitation, in one case by a factor of 12.

Early in the testing, it was observed that the carbon conversion rates were related to particle size as viewed by gross examination. As a result, select samples were submitted for size analysis. The carbon conversion rates of the three samples analyzed were not related solely to particle size as determined by small particle analysis. This discrepancy suggests that the conversion rate may be related instead to other physical parameters such as surface area and/or physical ash or residue formation or structure relative to the reactant species. Sample preparation will therefore be a necessary area of research in defining efficiencies for this system application.

Samples of the inert matrices (i.e., sand and silica gel) taken before and after processing were analyzed using x-ray diffraction and x-ray fluorescence. The results of these analyses indicated that the processing had not caused any significant morphological changes.

#### CONCLUSIONS

The results of this testing indicate that the agitated low-temperature plasma reactor system successfully converted carbon, hydrogen, and nitrogen to gaseous products at residence times that were approximately ten times shorter than those achieved by stationary processing. The inorganic matrix present was virtually unchanged by the processing technique. It is concluded that, at this stage of development, this processing technique is feasible for use as part of a CLP resource management system. Further investigation to optimize reactor configuration, energy requirements, and scale-up are warranted based upon the results of this work.

#### REFERENCES

1. "Application of Low-Temperature Plasma Reactors as a Resource Management Subsystem in Closed-Loop Processing Applications," Proposal submitted to NASA on August 13, 1987, by the Advanced Technologies Group of the University of North Dakota Energy Research Center.
2. Hetland, M.D., Rindt, J.R., Jones, F.A., and Sauer, R.S., "Further Investigation of the Feasibility of Applying Low-Temperature Plasma Technology to a Closed-Loop Processing Resource Management System," Final report submitted to NASA, Contract No. NASW-4455, Project No. 10-49501 (April 1990).

TABLE 1

SUMMARY OF CONVERSION AND SOLUBILITY DATA FROM BOTH  
CURRENT AND EARLIER WORK (REF. 1)

	Feces (earlier work)	Feces	Sunflower (earlier work)	Sunflower	Low-Ash Carbon	High-Ash Carbon	Plastic (earlier work)	Plastic
% Residue <sup>a</sup>	31.00	29.82	18.52	14.84	4.24	29.85	1.40	<1.00
Time (hr in plasma)	22 ± 1	3 ± 0.1	24 ± 4	2 ± 0.1	3 ± 0.5	3 ± 0.5	48 ± 6	8 ± 2
CHN Analysis of Residue <sup>b</sup>								
% Carbon	5.205 ± 0.145	BDL <sup>d</sup>	5.220 ± 0.27	BDL	BDL	BDL	---	BDL
% Nitrogen	0.885 ± 0.045	BDL	0.440 ± 0.04	BDL	BDL	BDL	---	BDL
% Hydrogen	1.380 ± 0.06	BDL	1.655 ± 0.095	BDL	BDL	BDL	---	BDL
% Conversion <sup>c</sup>	98.39	100 <sup>e</sup>	99.03	100	100	100	98.60	100
% Solubility, H <sub>2</sub> O	32.33	---	82.36	84.09	46.67	16.67	---	---
% Solubility, 6M HCl	73.86	---	---	94.44	70.83	22.22	---	---

<sup>a</sup> Based on wt. of residue out of plasma reactor/wt. of samples into reactor

<sup>b</sup> Standard CHN on a Control Equipment Corp. unit

<sup>c</sup> Based on 1- residue - carbon wt. in residue - non-organic wt.

sample wt. - non-organic wt.

<sup>d</sup> Below detectability limits

<sup>e</sup> None detected; i.e., in/out indicated 100% loss

<sup>f</sup> Not enough sample for analysis

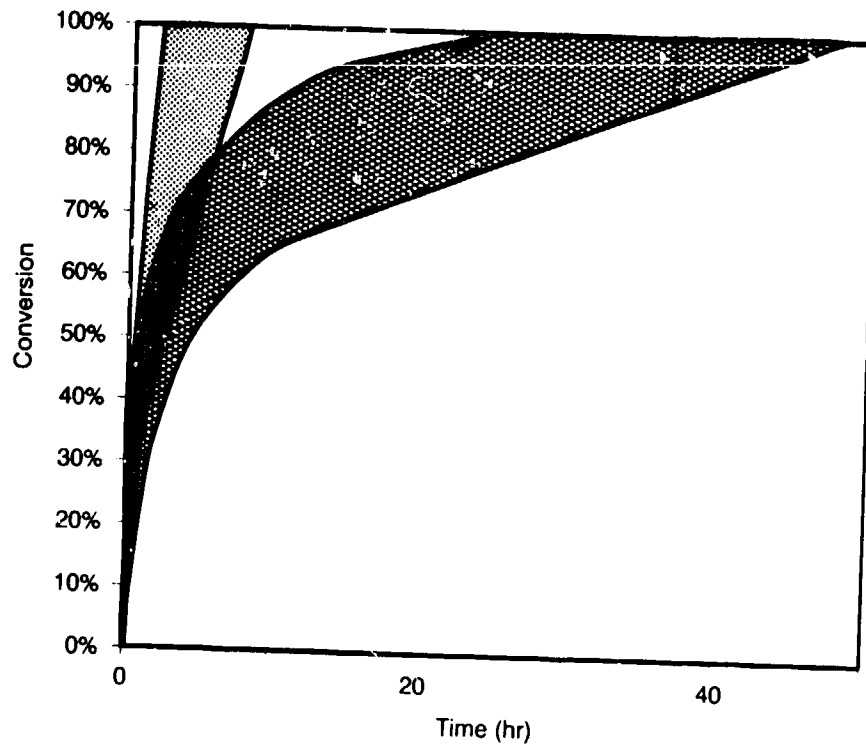


Figure 17. Ranges of residence times required for complete carbon conversion.