

FINAL TECHNICAL REPORT
September 1, 2004, through June 30, 2006

Project Title: **COAL-PREP WASTES IN CEMENT MANUFACTURING –
COMMERCIALIZATION OF TECHNOLOGY**

ICCI Project Number: 04-1/4.1A-2
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ABSTRACT

CTLGroup worked with several cement plants and Illinois mines to commercialize the use of coal prep-waste as a partial fuel in cement manufacturing. As reported earlier, a successful demonstration was carried out at Buzzi Unicem's cement plant in Greencastle, Indiana, using Peabody's Randolph prep-waste. As the next logical step, CTLGroup pursued the plant to commercialize the technology. However, citing uneconomic shipping cost, the plant declined to participate. Two approaches followed 1) to find prep-waste sites close to cement plants, or 2) to find cement plants close to the prep-waste sites. Carrier Mills, Marissa, Murdock, and Middle Grove were identified as the potential prep-waste sites; and Buzzi Unicem's Cape Girardeau, Oglesby, and Greencastle; Lafarge's Joppa; and Illinois's LaSalle were identified as the partnering cement plants.

CTLGroup approached Cape Girardeau and Joppa cement for the use of Marissa and Carrier Mills prep-wastes. These prep-wastes were compatible with the process of both cement plants. However, Marissa dropped out because of difficulties in hauling the material off their site; the Carrier Mills was also unable to obtain the processing permit. Consequently, the Murdock site was explored. Cape Girardeau plant agreed to participate in the pre-commercialization program. More than 100 tons material was transported to the plant. The material was blended with the regular fuel at 1:4 ratio and directly introduced into both the precalciner and rotary kiln. The overall operation remained stable and realized noticeable energy and environmental benefits. The plant expressed interest in the material and considered using it after their contract with the current fuel supplier expires. However they expressed concern over long shipping distance and preferred a site closer to their plant. CTLGroup is in touch with Carrier Mills site to this regard.

We contacted Buzzi Unicem to reconsider prep-waste use at Greencastle (and at Oglesby). We also contacted Illinois plant at LaSalle, and Lafarge at Joppa; they expressed genuine interest. However, because of prior commitment to fuel suppliers, they deferred the use of prep-waste. We expect that the cement plants will ultimately require an economically viable alternative fuel. Therefore CTLGroup intends to keep the contacts with the cement plants and mine sites ongoing – and plans to notify the ICCI and DCEO for appropriate response should an opportunity arrive. CTLGroup also believes that in order to expedite the commercialization of the prep-waste technology, the local and state governments could pass legislation to encourage the waste and by-product users with economical incentives.

EXECUTIVE SUMMARY

Following the successful demonstrations at the Buzzi Unicem's Greencastle cement plant (prior to the start of this project), CTLGroup vigorously pursued for the commercialization of coal prep-waste technology in cement manufacturing. We held a series of discussions with personnel from Greencastle cement plant and the Peabody's Randolph mine, along with ICCI personnel to address the underlying logistical issues. The talks could not move forward because of the economic issues involving costly transportation of the material.

This prompted CTLGroup to pursue a dual approach of locating potential prep-waste sites close to cement plants and finding cement plants close to the prep-waste sites. CTLGroup approached the Illinois Department of Natural Resources to identify potential coal prep-waste sites within Illinois. Sites near Carrier Mills, Murdock, Middle Grove, and Marissa were short-listed; samples were collected from these sites and analyzed. The data on fuel value, sulfur, and ash varied with sample location, select data (dry basis) are shown below.

	Murdock	Marissa	Middle Grove	Carrier Mills
Btu/lb range	9717 – 11135	7561 – 10408	4856 – 7690	9458 – 9804
Sulfur, % range	1.86 – 2.11	2.68 – 3.56	2.65 – 6.69	0.84 – 0.86
Ash, % range	14.28 – 23.89	26.22 – 43.92	41.97 – 60.98	12.41 – 14.91

The cement plants that could partner the commercialization program were Buzzi Unicem's Cape Girardeau, Oglesby, and Greencastle; Lafarge's Joppa; and Illinois's LaSalle.

CTLGroup contacted both the Cape Girardeau and Joppa cement plants and discussed implementation of prep-wastes from Marissa, and Carrier Mills. The materials from both sites had reasonably high fuel values, and despite high sulfur contents in Marissa prep-waste (see table above), were compatible with the cement plants' manufacturing process. After months of discussions and efforts, both Marissa and Carrier Mills, dropped out of the program. Marissa cited administrative difficulties in hauling the material out of their site; and the Carrier Mills was unable to obtain the site permit to process the material.

Consequently, the use of prep-waste from the Murdock site was explored. The prep-waste had around 27% moisture, 8200 Btu/lb fuel value, and <2% sulfur. The mean particle size of the prep-waste was approximately # 50-mesh. All cement plants expressed interest, but only Cape Girardeau agreed to participate within the project deadline. Several hundred tons of material was dredged from the site and spread for air-drying until the moisture was close to 20%. The material was loaded into trucks and shipped to Cape Girardeau plant.

The material was blended with the regular plant fuel (coal) at 1:4 ratio and simultaneously introduced into the precalciner and the rotary kiln. The emissions including the mercury levels were monitored and compared with the levels recorded before the demonstration. The overall operation during the demonstration remained stable and trouble-free. The 20% fuel substitution by prep-waste resulted in a 21% saving in purchased fuel. During

the demonstration, no change was observed in the operation and environment; the emission levels were normal, however, the mercury level was reduced by an order of magnitude. No change was seen in the product properties. It must also be mentioned that this demonstration was significantly more beneficial than the Greencastle one (conducted previously); see a summarized comparison below:

Parameters	Greencastle	Cape Girardeau	Comments
Prep-waste processing	Processing required	Used as is	Advantage Cape G.
Use level	10%	20%	Advantage Cape G.
Introduction at	Precalciner only	Precalciner & kiln	Advantage Cape G.

Following the successful demonstration, CTLGroup discussed the commercialization option with Cape Girardeau. The plant expressed interest in considering the material after their contract with the current fuel supplier is over. However they expressed concern over the long shipping distance and preferred a site closer to the plant. CTLGroup is discussing this option with the Carrier Mills site, which is only 70 miles away from the plant.

The demonstration at the Cape Girardeau also prompted us to contact Buzzi Unicem to consider using the prep-waste at their Greencastle and Oglesby cement plants which are respectively 70 and 135 miles away from Murdock; we also contacted Illinois plant at LaSalle, and Lafarge at Joppa for the prep-waste use. Prep-waste samples were sent to them upon request. Two of the plants expressed concern over the ash content and the volatile matters in the prep-waste. Despite these concerns, which can be adequately addressed by adjusting the substitution level of the prep-waste and reformulating the raw mix, the plants were interested in the prep-waste. However, because of their pre-commitment to the fuel supplier, they deferred the use to an opportune time in future.

Our discussions with the cement plants and prep-waste suppliers also raised the existence of the same old perceptions – the cement plants contend that they are serving the prep-waste suppliers by using their ‘waste,’ hence they are entitled to economic incentives, whereas the prep-waste suppliers state that, since they are not getting the worth of their energy-laden material, it is not worth their effort to provide their material to cement plants.

CTLGroup believes that although the current climate may not be conducive to the cement plant and prep-waste economics, this is most likely transitory. The cement plants have genuine interest in this material; at an opportune time their need may change whereby they would require an economically viable fuel supplement. Therefore, CTLGroup intends to keep the contacts ongoing with the cement plants and the prep-waste sites. The approach may also require economic incentives from ICCI and DCEO for system modification to improve material processing. Both the Carrier Mills and Murdock sites were made aware of the DCEO incentive plan. For an economically viable shipping, backhauling of CKD from cement plant was also considered, which, when put in place, can make prep-waste even more economically attractive.

CTLGroup also believes that in order to expedite the commercialization of the prep-waste technology, the local and state governments could pass legislation to encourage the waste and by-product users with economical incentives. CTLGroup, ICCI, and DCEO should jointly consider pursuing this avenue.

OBJECTIVES

The objectives of this project were to commercialize the use of Illinois coal prep-wastes as a fuel supplement in cement manufacturing and successfully realize economical, environmental, and product benefits. The objectives also included the generation of a widespread interest of this technology amongst cement plants and identify logistic issues that might hinder the full-scale implementation of the technology. Based on preliminary calculations and approximations, it was anticipated that:

- A 15-20% substitution rate of prep-waste with regular fuel in the four Illinois cement plants with a combined capacity of 3.2 million tons/year, could utilize over 150,000 tons of prep-waste each year as a fuel supplement to manufacture commercial cement.
- The use of prep-waste will not adversely impact the emissions at cement plant.
- This "waste-to-energy" approach would generate a saleable product while significantly reducing wastes and related environmental stresses in Illinois.
- The quality of the cement, as judged by testing using methods outlined in ASTM C 150, should be comparable to that produced using the original fuel and raw materials.

The role of CTLGroup was the technology developer and promoter, whereas those of ICCI and DCEO were of as-needed technology promoters. CTLGroup involvement as technology developer included offsite/onsite technical support and expert assistance to both cement manufacturers and prep-waste producers to facilitate the use of large volumes of prep-waste in cement plants. The plants are expected to run a smooth, reliable, economical, environmentally safe, and trouble-free operation. Another aspect of CTLGroup's onsite and offsite support was to organize team meetings/conference calls on as-needed basis to resolve impending issue regarding the technology commercialization. The role ICCI and DCEO were to assist, upon request, in addressing material- and site-specific issues, and consider potential incentives for system modifications.

INTRODUCTION AND BACKGROUND

Within the State of Illinois, several million tons of coal prep-wastes are annually generated at coal mines. Only a portion of these wastes is used as the mine backfills, while the bulk is either ponded or landfilled. At the same time, cement plants within the vicinity have a need for alternative cost-effective fuels and process improvements. Since the prep-wastes often contain substantial proportions of coal, they have the potential for use as a supplementary fuel. This has been demonstrated in CTLGroup's previous ICCI-sponsored industrial-scale project regarding the feasibility of using coal prep-waste as a fuel component.

This report outlines CTLGroup's continued effort on the use of Illinois coal prep-waste as an economical fuel supplement in the follow up demonstration at a cement manufacturing plant. The report also discusses the implementation aspect of technology and the logistical issues encountered during the commercialization efforts.

EXPERIMENTAL PROCEDURE

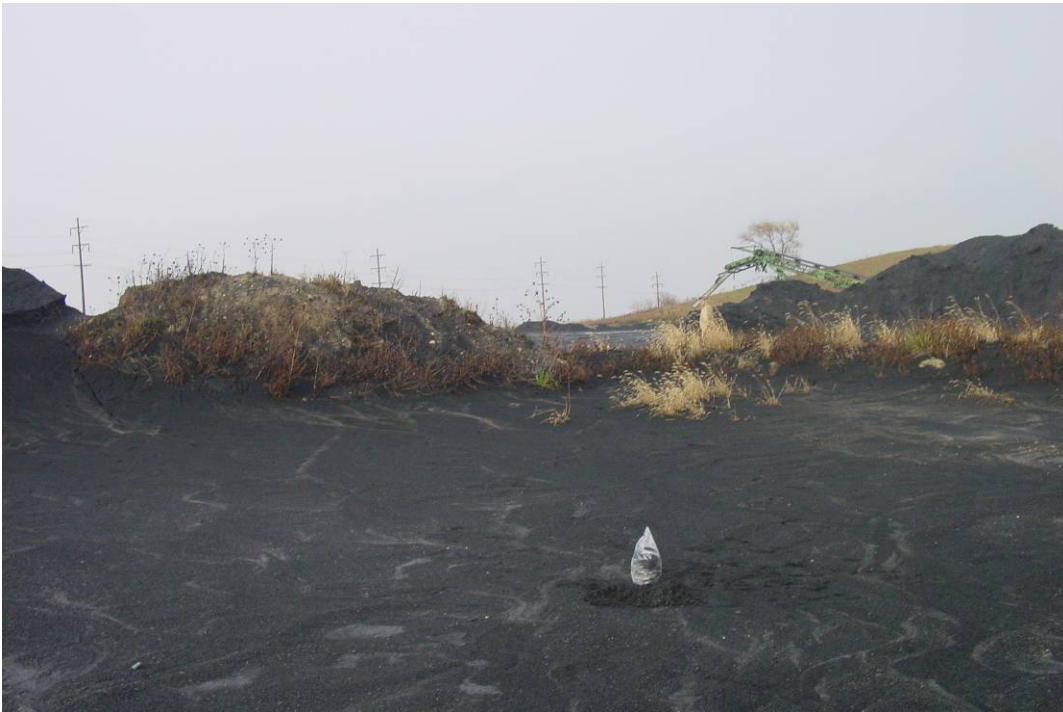
The testing, characterization, and evaluation of the coal prep-waste samples (collected from mine sites), the raw materials, and clinker samples (collected from cement plant during demonstration) were carried out at the CTLGroup facilities. Preparation of cements was carried out by using an 8-pound ball mill. The testing procedures used for oxide analyses, clinker compound analyses, and phase distribution included X-ray fluorescence (XRF), X-ray diffraction (XRD), and reflected light microscopy. Determination of fuel value and emission related species in the prep-wastes were conducted by differential scanning calorimetry (DSC) technique. Testing of stack emissions including mercury were contracted to outside sources.

RESULTS AND DISCUSSION

The project started with identifying mine site and potential partnering cement plants. CTLGroup contacted Illinois Department of Natural Resources (DNR) to identify several potential coal prep-waste sites.

Task 1. Identifying Prep-Waste Sites and Partnering Cement Plants

The short listed coal prep-waste sites were Marissa and Carrier Mills (both in Southern Illinois); and Middle Grove and Murdock (both in Central Illinois); some of the selected sites are shown in Figure 1a, b, and c.



a) Murdock, Central Illinois



b) Carrier Mills, Southern Illinois



c) Marissa, Southern Illinois

Figure 1. Coal Prep-Waste Sites as Potential Participants in Demonstration

Likewise, the most probable participating cement plants selected for the commercialization program were Buzzi Unicem's Greencastle, Indiana; Cape Girardeau, Missouri; Oglesby, Illinois; and Lafarge's Joppa, Illinois; and Illinois's LaSalle, Illinois. The locations of mines with respect to the cement plants are given in Table 1. Plant capacities, process configurations, and proposed mode of prep-waste introduction, are also given in the table.

Table 1. Cement Plants Capacities, Configurations, and Prep-Waste Locations and Distances

Cement plants	Plant capacity Tons/year	Process configuration	Proposed mode of introduction	Prep-waste site, and distance (miles)
Oglesby, IL	600,000	Long dry	Mid-kiln	Middle Grove (75)
Greencastle, IN	1,300,000	Precalciner	Precalciner	Murdock (70)
Cape Girardeau, MO	1,300,000	Precalciner	Precalciner	Carrier Mills (70)
Cape Girardeau, MO	1,300,000	Precalciner	Precalciner	Marissa (80)
Joppa, IL	1,060,000	Long dry	Mid-kiln	Sahara (50)

Out of these, Greencastle has been identified because of its participation in the short-term demonstration with the Peabody's Randolph prep-waste. In light of Buzzi Unicem's interest in the technology, their other plants were also short listed for inclusion in the program.

Task 2. Mapping, Sampling, and Characterization of Prep-Wastes

The Murdock, Carrier Mills, Middle Grove, and Marissa sites were visited and several sampling locations were identified. The prep-waste samples were taken from those locations for analyses on physical, chemical, thermal, fuel, and emission-related properties. The typical analyses data of the prep-waste samples on dry basis are given in Tables 2, 3, 4, and 5.

Table 2. Marissa Prep-Waste Samples

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Btu/lb	8889	7561	10408	8876	9449
Sulfur, %	2.93	2.68	2.91	3.45	3.57
Ash, %	35.03	43.92	26.22	34.12	31.34

Table 3. Carrier Mills Prep-Waste Samples

	Sample 1	Sample 2	Sample 3	Sample 4
Btu/lb	9804	9458	9742	9534
Sulfur, %	0.85	0.84	0.86	0.85
Ash, %	13.83	14.77	14.91	12.41

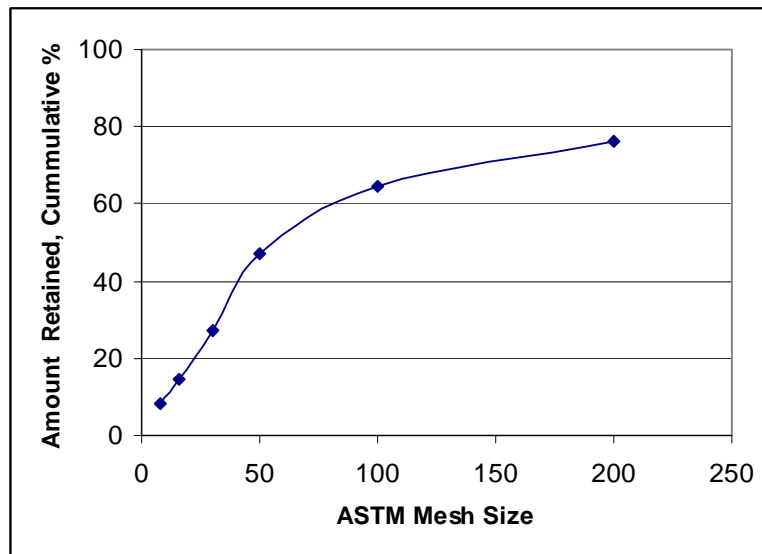
Table 4. Murdock Prep-Waste Samples

	Sample 1	Sample 2	Sample 3	Sample 4
Btu/lb	9877	11135	10305	9717
Sulfur, %	2.10	1.86	2.11	2.03
Ash, %	20.01	14.28	19.61	23.89

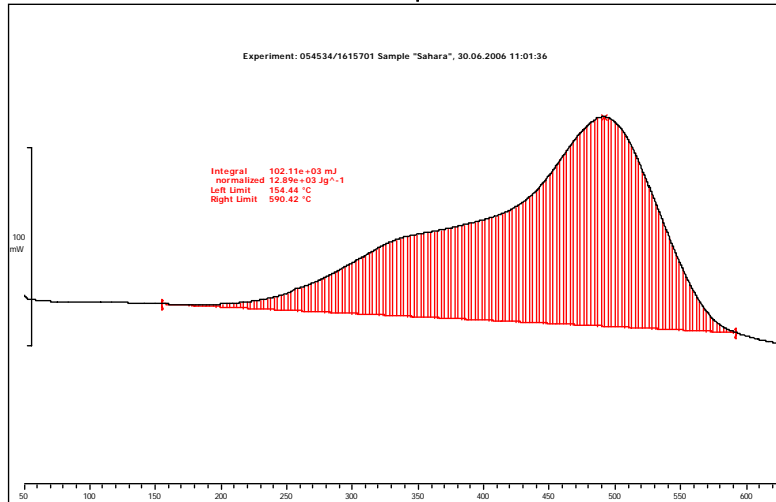
Table 5. Middle Grove Prep-Waste Samples

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Btu/lb	5824	7423	7690	4882	4856
Sulfur, %	2.65	3.67	4.06	6.69	6.43
Ash, %	54.80	43.95	41.97	59.68	60.53

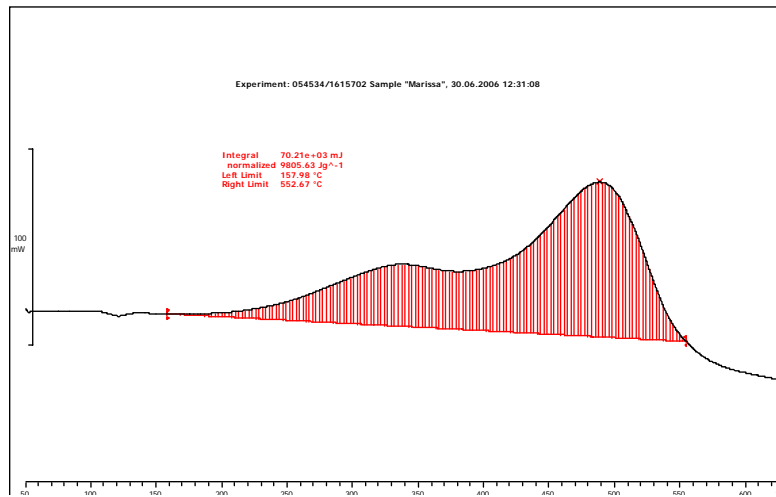
The particle size distribution of the Murdock prep-waste, expressed as the cumulative amount retained, is shown in Figure 2. The data indicate an average mean size of the material to be around 50 mesh.

**Figure 2. Particle Distribution of Murdock Prep-Waste Showing 50-Mesh as the Mean Size**

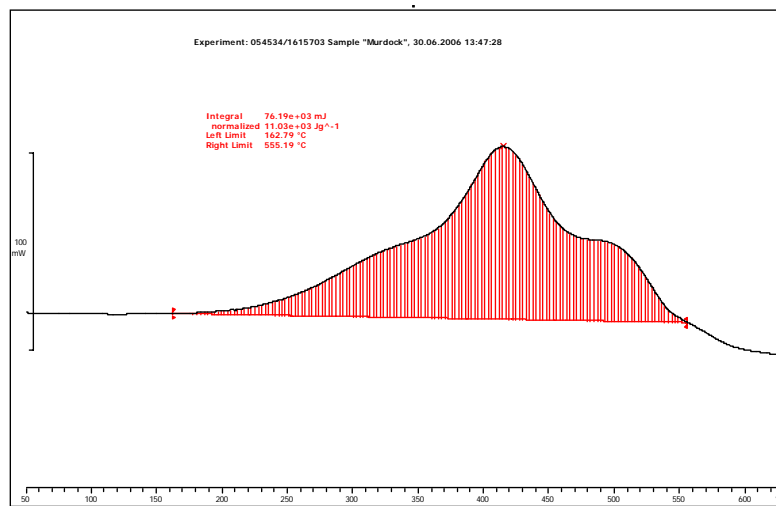
DSC plots on the representative prep-waste samples are shown in Figures 3a, b, and c. The presence of large endothermic humps between 250°C and 600°C peaking around 500°C confirms the presence of substantial heat value in the material.



a) Carrier Mills prep-waste



b) Marissa prep-waste



c) Murdock prep-waste

Figure 3. DSC Plots of Prep-Waste Samples Showing Heat Values

Site Specific Scenarios: As can be seen from the data, except for the Middle Grove, prep-waste from all other sites have reasonably high fuel (>9000 Btu/lb) and low sulfur values (<2.5%). Middle Grove prep-waste was disregarded because of low heat, inconsistency in composition, and high sulfur contents. As the discussion on excavation and transportation of prep-waste proceeded, the Marissa cited potential difficulties in shipping the material and declined to participate further. Later on the Carrier Mills mine was not able to obtain the processing permit for their site. This left us working only with the Murdock site.

Task 3. Bulk Preparation and Processing of Prep-Wastes

Of all the possible teams of mine site and cement plants listed in Tasks 1 and 2, CTLGroup preferred the team of the Murdock site and Buzzi Unicem plant in Cape Girardeau. This team had the potential of several advantageous options, which included 1) the use of the as-received prep-waste without any pre-blending with coal, as was done previously with the Randolph prep-waste during the demonstration at Greencastle, 2) exercising the maximum allowable use of 20% replacement of their regular fuel as opposed to 12% in the previous demonstration, and 3) using the prep-waste/fuel blend as the primary fuel in both the precalciner and the rotary kiln.

A large amount of prep-waste was dredged from the site and hauled to an open area to facilitate air-drying. Once the material reached an acceptable dryness (about 20% moisture) and was sufficiently free-flowing and blendable, more than 100 of tons prep-waste was shipped by trucks to the cement plant. Prep-waste preparation, loading, and shipping from Murdock, and consequent unloading at Cape Girardeau cement plant are shown in Figure 4a, b, c, and d.



a) Prep-waste dredging and spreading



b) Prep-waste hauling



c) Prep-waste loading into trucks



d) Prep-waste shipping

Figure 4. Prep-Waste Processing, Loading, and Shipping from Murdock

Several trucks were loaded and shipped to the Buzzi Unicem's cement plant in Cape Girardeau, MO. The material was unloaded in the open (Figure 5) and left overnight for further air-drying. The air-drying, hauling, and shipping operation was timed for dry weather to avoid rain. The prep-waste delivered to the plant had the proximate analysis as shown in Table 6.

Table 6. Proximate Analysis of Murdock Prep-Waste Delivered to Cape Girardeau

	Moisture, %	Btu/lb	Sulfur, %	Ash, %	Fixed C, %	Vol. matter, %	Cl ₂ , %	Hg, ppm
As Received	23.2	7820	1.31	15.99	32.67	28.14	0.07	0.12
Dry Basis	-	10180	1.71	20.82	42.54	36.64	0.09	0.148

The prep-waste was blended with the regular plant fuel at 1:4 ratio (20% prep-waste and 80% plant fuel), and placed in the fuel stored storage. During the demonstration, the fuel blend was simultaneously introduced into the precalciner (Figure 6) as well as into the rotary kiln (Figures 7 and 8) as the primary fuel.



Figure 5. Prep-Waste (dark piles) Unloaded in Open at Cape Girardeau Plant for Air-Drying



Figure 6. Prep-Waste and Regular Fuel Blend Introduced into Precalciner



Figure 7. Prep-Waste/Regular Fuel Blend Introduced (yellow pipe) into Kiln



Figure 8. Rotary Kiln at Cape Girardeau Cement Plant

Task 4. Long-Term Demonstration at the Cement Plant

Identifying the Method of Prep-Waste Introduction into Kiln

Cape Girardeau is a short dry kiln with a preheater/precalciner configuration. As mentioned in Task 3, the plant maximized the use of prep-waste, by substituting it for 20% of their regular fuel (coal). The prep-waste was blended with the regular fuel prior to its introduction into the cement kiln system. A calculated amount of blend was simultaneously introduced into the system by:

- 1) Injecting into the precalciner, and
- 2) Injecting into the burning zone from the front end of the rotary kiln.

Kiln Burn and Operational Observations

During the demonstration, a number of parameters were monitored at the plant as summarized bellow.

Stack Emissions: First and foremost were the monitoring of stack emissions such as the O₂, CO, NO, NO₂, SO_x, NO_x, particulate matters, mercury, and stack opacity or detached plumes. Baseline values for these parameters were taken with the regular fuel as a standard of comparison. Data are summarized in Tables 7 and 8:

Table 7. Emission Data for Prep-Waste/Fuel Blend

Parameter	Units	Range During Test	Average
O ₂ ⁽¹⁾	(% by volume)	10.1 to 11.8	11.24
CO ⁽¹⁾	(ppm)	1100 to 3390	1492
NO ⁽¹⁾	(ppm)	48 to 254	162
NO ₂ ⁽¹⁾	(ppm)	0 to 3	0.94
SO ₂ ⁽¹⁾	(ppm)	0 to 32	0.37
Particulate Matter ⁽²⁾	(lb/dscf*)	1.4 x 10 ⁻⁰⁶ to 3.73 x 10 ⁻⁰⁶	2.36 x 10 ⁻⁰⁶
Particulate Matter ⁽²⁾	(lb/hour)	22.6 to 66.2	38.6
Mercury ⁽²⁾	(lb/dscf)	17.98 x 10 ⁻¹⁰ to 1.76 x 10 ⁻⁰⁹	1.21 x 10 ⁻⁰⁹
Mercury ⁽²⁾	(lb/hour)	0.013 to 0.029	0.020

(1) Data collected continuously during burning of the test fuel

(2) Average is based on values from three test runs (three data points)

* Dry standard cubic feet

Table 8. Emission Data for Regular Plant Fuel

Parameter	Units	Range During Test ⁽¹⁾	Average ⁽²⁾
O ₂ ⁽¹⁾	(% by volume)	9.7 to 12.1	11.41
CO ⁽¹⁾	(ppm)	15 to 3019	940
NO ⁽¹⁾	(ppm)	15 to 216	157
NO ₂ ⁽¹⁾	(ppm)	0 to 2	0.6
SO ₂ ⁽¹⁾	(ppm)	0 to 186	79
Particulate Matter ⁽²⁾	(lb/dscf)	2.15 x 10 ⁻⁰⁶ to 2.3 x 10 ⁻⁰⁶	2.2 x 10 ⁻⁰⁶
Particulate Matter ⁽²⁾	(lb/hour)	34.5 to 32.5	33.5
Mercury ⁽²⁾	(lb/dscf)	3.28 x 10 ⁻⁰⁸ to 3.69 x 10 ⁻⁰⁸	3.49 x 10 ⁻⁰⁸
Mercury ⁽²⁾	(lb/hour)	0.49 to 0.56	0.53

(1) Data collected continuously during burning of the regular fuel

(2) Average is based on values from two test runs (two data points)

It may be noted that the average of most emissions recorded prep-waste demonstration are close to the ones recorded with regular fuel. However, both SO₂ and mercury levels dropped significantly; mercury dropped by at least an order of magnitude. The CO level rose about 50% during the use of prep-waste blend fuel.

Kiln Parameters Observed

Several key parameters were observed while the kiln operation was in progress. Particular attention was given to the operational parameters related to prep-waste fuel supplement benefiting the overall operation as compared to those prior to the demonstration.

Production Rate, Fuel Rate, Kiln RPM. Given that the production rate is directly related to the kiln feed rate, it was noted that the fuel modification did not impart any adverse change as the feed rate during the demonstration remained almost the same as was prior to it. The data on the kiln feed rate, the fuel rate, and RPM during and before the demonstration are shown in Table 9.

Table 9. Feed Rate, Fuel Rate, and Rotary Kiln RPM*

Parameters	Kiln feed rate, tons/day	Fuel rate, lbs/min	Kiln rotation, RPM
Before	296	316	203
During	294	327	201

* RPM = Revolutions per minute

It may be noted that about 4% additional fuel was required to maintain the production level during the demonstration; this is understandable as the fuel value of the prep-waste

was marginally lower than the regular plant fuel. Nonetheless, during the demonstration the kiln operation ran smoothly.

Kiln Feed Temperature, and Burning Zone Temperature: Kiln feed temperature as it exits the precalciner and enters the rotary kiln, shows no variation prior to and during the demonstration; data on kiln feed temperature are shown in Table 10.

Table 10. Kiln Feed Temperatures

Parameters	Temperature, °F (°C)
Before	1607 (875)
During	1610 (877)

Since no noticeable variation in the kiln feed temperature was observed in the precalciner, no adjustment was required to accommodate prep-waste as the kiln fuel; the burning zone temperature also remained unchanged during the demonstration. This can also be inferred from the identical morphology of belite crystals in clinkers collected before, during, and after the demonstration. All clinkers show typical round belite crystals in photomicrographs (see Figure 8), suggesting that the kiln ran without any abnormal temperature variations during the demonstration.

Fuel Rate/Fuel Consumption: As noted above, the fuel rate/consumption during the demonstration was similar to the one prior to it, and the kiln temperature was also not adjusted during the demonstration, therefore, a rough estimation of saving on purchased fuel can be made assuming that an overall 20% fuel substitution was made with the prep waste; an estimation of fuel saving was made as follows:

Regular fuel	= 316 lb/min	
Total fuel during demonstration	= 327 lb/min	
Regular fuel in demonstration	= 80% of 327	= 261.6 lb/min
Regular fuel savings	= 327 – 261.6	= 65.4 lb/min
Net Fuel Savings, %	= (65.4/316) x 100	= 20.7%

Overall Operational Observations

During the demonstration of using the coal-prep waste, the operation ran smoothly. There were no problems of prep-waste delivery and blending with the regular plant fuel, plugging of fuel lines, or abnormal temperature profiles of the precalciner and the rotary kiln. There were no emission problems with respect to SO_x, NO_x, VOC, THC, particulate matters, and mercury; or environmental problems with respect to stack opacity or detached plumes. The demonstration was particularly successful as the fuel economy showed tangible improvement.

Task 5. Clinker Characterization and Evaluation of Cement

The clinkers and cements produced during the demonstration were characterized for physical and chemical properties. Clinkers were examined for mineralogical composition and major phase distribution whereas cement produced there from were tested and evaluated for compliance with the ASTM C 150 specification.

Clinker Characterization

The clinkers produced before, during, and after the demonstration were analyzed for quantitative determination of oxides and of Bogue compounds. Data are shown in Tables 11 and 12.

Table 11. Clinker Composition Before, During, and After Demonstration, wt. %

Analyte	Before	During	After
SiO ₂	21.81	21.44	21.70
Al ₂ O ₃	4.67	4.66	4.69
Fe ₂ O ₃	3.21	3.25	3.35
CaO	64.05	63.86	63.24
MgO	4.14	4.30	4.40
SO ₃	0.75	0.84	0.75
Na ₂ O	0.13	0.14	0.12
K ₂ O	0.49	0.60	0.56
TiO ₂	0.34	0.35	0.33
P ₂ O ₅	0.09	0.09	0.09
Mn ₂ O ₃	0.07	0.07	0.08
SrO	0.08	0.08	0.08
Cr ₂ O ₃	0.01	0.01	0.01
ZnO	0.02	0.02	0.02
Ignition Loss (L.O.I)	0.40	0.47	0.85
Free lime	1.40	1.21	0.97
Alkalies as Na ₂ O	0.46	0.54	0.49

Table 12. Bogue Composition in Clinkers Before, During, and after Demonstration, wt. %

Phase	Before	During	After
C ₃ S	57	59	54
C ₂ S	20	17	22
C ₃ A	7	7	7
C ₄ AF	10	10	10

Clinkers were further tested by X-ray diffraction (XRD) to identify mineralogical composition, and examined by reflected microscopy for the distribution of the major clinker phases. The data from Bogue composition and the subsequent XRD analysis (Figure 9) confirmed the presence of major C₃S, C₂S, C₃A, and C₄AF phases in clinkers.

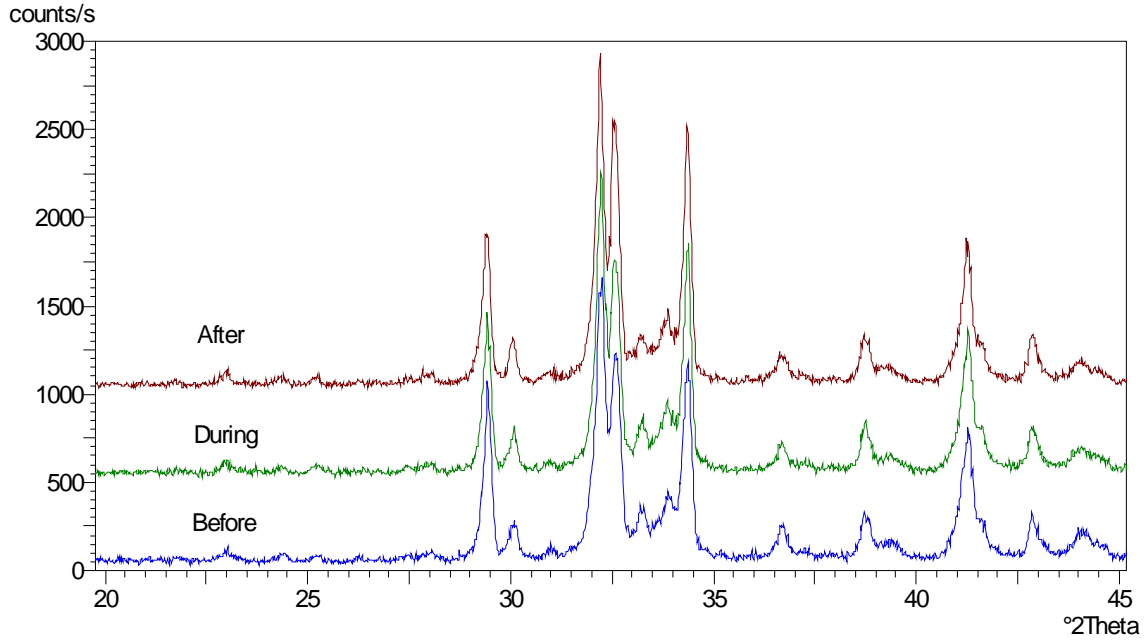
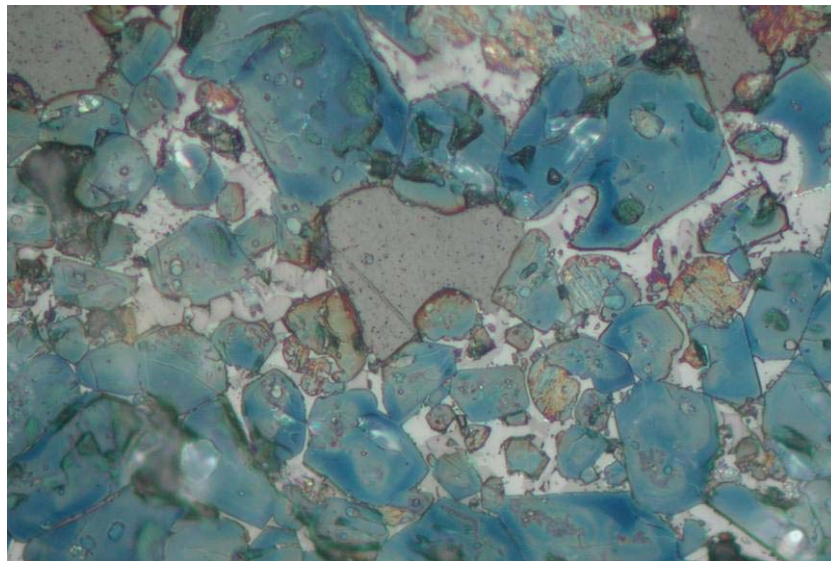


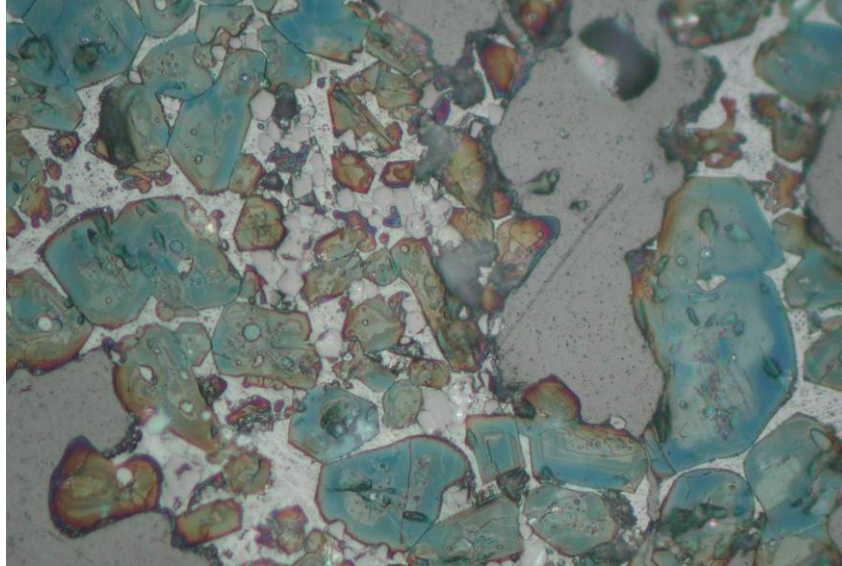
Figure 9. XRD Patterns of clinker produced before, during, and after the demonstration

Absence of free lime peaks in all clinkers including the demonstration clinkers can be attributed to an improved reactivity of lime in the raw mix with the prep-waste ash rendered by its fine and glassy particles.

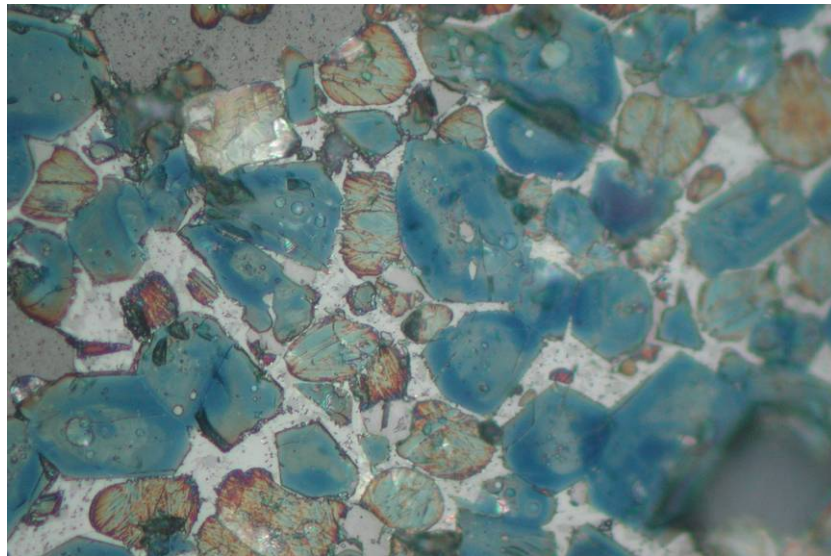
Polished sections of the clinker were examined by reflected light microscopy to determine the relative quantities and distributions of the major phases. The photomicrographs of the clinkers produced before, during, and after the demonstration are shown in Figure 10a, b, and c.



a) Clinker before demonstration (with regular fuel)



b) Clinker during demonstration (with prep-waste/fuel blend)



c) Clinker after demonstration

Figure 10. Clinker Photomicrographs – Magnification x 400

As mentioned earlier, the identical morphology, i.e., the size, shape, and distributions of crystals – particularly of the belite, which show typical round belite crystals, suggest that the kiln ran without any abnormal temperature variations during the demonstration. Presence of porosity also typified the clinkers and its predictability for ease in grinding.

The clinkers were interground with appropriate amount of plant gypsum to the normal plant fineness (about $350 \text{ m}^2/\text{kg}$). Laboratory-scale ball mill of an 8-lb capacity was used for grinding. Exactly an 8-lb load of previously crushed clinkers (using jaw and gyratory crushers) were used and time taken to achieve the required fineness was recorded for each clinker as in Table 13.

Table 13. Time to Reach Required Blaine Clinker Fineness, minutes

Parameter	Clinker Before	Clinker During	Clinker After
Grinding time required, min	65	63	65
Blaine fineness, m ² /kg	377	369	380

The demonstration clinker grindability was almost identical to those produced normally, and thus would not require additional energy for finished cement grinding.

Evaluation of Cement per ASTM C 150 Specification

Cements produced were tested for both the chemical and physical properties as per the ASTM C150 specification. Chemical compositions and Bogue analyses are given in Table 14.

Table 14. Chemical Composition and Computed Bogue Compounds of Cements, wt. %

Analyte	Cement Before	Cement During	Cement After
SiO ₂	20.78	20.45	20.54
Al ₂ O ₃	4.50	4.41	4.48
Fe ₂ O ₃	3.25	3.21	3.36
CaO	62.55	62.43	61.65
MgO	4.03	4.15	4.26
SO ₃	2.94	3.04	2.99
Na ₂ O	0.10	0.11	0.11
K ₂ O	0.47	0.54	0.55
TiO ₂	0.32	0.31	0.31
P ₂ O ₅	0.09	0.09	0.09
Mn ₂ O ₃	0.08	0.07	0.08
Cr ₂ O ₃	<0.01	0.01	0.01
ZnO	0.02	0.02	0.02
Ignition Loss	0.87	1.07	1.34
Alkalies as Na ₂ O	0.41	0.46	0.47
Insoluble Residue	0.23	0.17	0.49
Computed Bogue Compounds, wt. %			
C ₃ S	56	53	57
C ₂ S	17	19	17
C ₃ A	6	6	7
C ₄ AF	10	10	10

These cements were tested and evaluated in accordance with the ASTM C 150 specification for their physical properties. The data are shown in Table 15.

Table 15. ASTM C 150 Data of Cements

	Before	During	After	ASTM Limits
ASTM C 185 – Air content, %				
	8.3	6.8	7.8	12 (max)
ASTM C 204 – Fineness, air permeability (Blaine), m ² /kg				
	377	369	380	280 (min)
ASTM C 109 – Compressive strength, MPa (psi)				
3-day	29.4 (4260)	28.5 (4130)	25.4 (3690)	1800 (min)
7-day	37.1 (5380)	38.0 (5510)	33.5 (4860)	2800 (min)
28-day	53.6 (7780)	50.1 (7260)	47.6 (6900)	4060 (min)
ASTM C 191 – Vicat time of set, minutes				
Initial	120	130	135	45 (min)
Final	185	190	200	375 (max)
ASTM C 151 – Autoclave expansion, %				
	0.06	0.07	0.06	0.80 (max)

The results in Tables 14 and 15 clearly show that the properties of the cement produced during demonstration are comparable to those of the normally produced cement and are in compliance with the ASTM C150 chemical and physical specifications.

Task 6. Economic Outlook and Commercialization Scenarios

As briefly mentioned earlier, the principal benefits realized from the Cape Girardeau plant demonstration were 1) their acceptability of Murdock prep-waste with minimal material processing 2) maximizing the use of prep-waste 3) using the fuel-blend directly in the precalciner as well as in the rotary kiln. Based on these considerations CTLGroup discussed commercializing the prep-waste as a fuel supplement at the plant. The plant expressed keen interest in considering the material after their current contract with the fuel supplies is exhausted. However, citing high shipping costs involved between Murdock and Cape Girardeau, they preferred a closer prep-waste site. CTLGroup is touch with Carrier Mills (70 miles from the plant) to expedite their acquisition of permit for a beneficial use of their prep-waste.

Likewise, we also contacted cement plants close to the Murdock site; these included Buzzi Unicem's plants in Greencastle, Indiana, and in Oglesby, Illinois. The Illinois Cement plant in LaSalle, Illinois was also contacted. The Murdock prep-waste samples were sent to them upon request. Based on the high fuel value and favorable data from the Cape Girardeau demonstration, the plants also expressed interest in the material. However owing to their prior commitment to the fuel suppliers, they deferred the use of the prep-waste at present time.

Since the underlying logistical challenge to the implementation was the material economics, the scope of infrastructure improvements, either at the mine sites or at cement plants, was also discussed to facilitate material preparation, processing, and transportation. To this regard CTLGroup discussed the benefits of ICCI and DCEO economic incentive plan for infrastructure improvement with the prep-waste sites (Murdock and Carrier Mills), and cement plants within Illinois (Illinois, Oglesby, and Joppa) – should they engage in a long-term commitment to the prep-waste use. Also discussed was the possibility of backhauling CKD (cement kiln dust) from cement plants and its significance in subsidizing the prep-waste shipping cost. The parties were receptive to the propositions and were to look for an opportune time in future to consider them.

Task 7. Group Meetings

In order to address the scenarios outlined in Task 6, CTLGroup organized discussions with the key personnel from the potential cement plants and the Murdock prep-waste site to promote commercialization of the technology. Discussions with the following groups were conducted:

1. Buzzi Unicem's Cape Girardeau Plant, CTLGroup, Murdock and Carrier Mill Sites.

Following the successful Cape Girardeau demonstration, CTLGroup discussed with the plant personnel about the commercialization of prep-waste. The plant showed genuine interest in the prep-waste and expressed willingness to consider its use after their contract with the current fuel supplies is expired. However, due to high anticipated shipping costs, they suggested a closer prep-waste site. CTLGroup contacted Carrier Mills (70 miles from the plant) to expedite their permit acquisition to process their prep-waste. Our discussions with them are ongoing.

2. Buzzi Unicem's Greencastle and Oglesby Plants, CTLGroup, and Murdock Site:

Because of the beneficial demonstration of the Murdock prep-waste, CTL approached Buzzi Unicem for reconsidering its use at Greencastle, and also in Oglesby. Consequently, prep-waste sample were dispatched to them for evaluation. Despite a concern about the prep-waste ash content, they express an interest in the material. However, they deferred the use because of the pre-commitment to the existing fuel supplier. CTLGroup discussions with them are ongoing.

3. Illinois Cement plant, CTLGroup, and Murdock Site.

Again with the backdrop of the Cape Girardeau demonstration, CTLGroup approached Illinois Cement and discussed the use of Murdock prep-waste at their LaSalle plant. Upon request, a prep-waste sample and its ash analysis were furnished; see Table 16 for analysis. There was concern about the moisture and ash content of the prep-waste. We believe the moisture can be reduced by air-drying, and ash can be consumed by adjusting the mix design. CTLGroup is discussing with the plant to resolve the issue.

Table 16. Prep-Waste Ash Analysis, wt. %

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	SrO	ZnO	L.O.I.
55.90	22.77	10.61	2.04	1.23	1.41	0.32	2.58	1.09	0.23	0.03	0.03	0.20

4. Lafarge Joppa, CTLGroup, Murdock and Carrier Mill site personnel.

CTLGroup also discussed with Lafarge regarding the use of Murdock prep-waste at Joppa plant. Upon request, a prep-waste sample and its proximate analysis were furnished to the plant. Data on the proximate analysis is already documented in Table 6. They expressed concern about the volatile matter of the prep-waste. Since their plant has the mid-kiln injection system, the prep-waste may cause higher emission levels. Also of concern was the long shipping distance. Consequently, CTLGroup is discussing with Carrier Mills site (50 miles from the plant) to expedite their acquisition of the permit to process their prep-waste for use at Joppa. Our discussions with the cement plant and Carrier Mills are ongoing.

Task 8. CTLGroup Technical Support

The objective of this task was to provide technical support on an as-needed basis to both cement and prep-waste plants to facilitate the commercialization of the technology. As part of the offsite technical support, CTLGroup provided data on prep-waste characterization and consistency in terms of moisture level, fuel, volatile, and ash content. CTLGroup also provided specific data on the prep-waste ash and volatile matter to Illinois and Greencastle; and Lafarge cement plants respectively. The testing of clinkers and cements was also part of offsite support. Planning with the subcontractors, shipping material, and assisting with the monitoring of stack emissions during the demonstration was part of the onsite support. The scope of work for this task was relatively undefined but was within the competence of CTLGroup expertise.

CONCLUSIONS AND RECOMMENDATIONS

The industrial-scale demonstrations at two cement plants including the present one at Cape Girardeau (and the previous one at Greencastle plant) using separate prep-wastes have shown that cement manufacturing can be employed in consuming large volumes of prep-waste as a fuel supplement. The demonstrations provide a viable market for the previously discarded coal prep-waste with tangible energy and environmental benefits to both coal mines and cement industry.

It may be pointed out that the demonstration at Cape Girardeau was more beneficial than the one conducted at Greencastle. In that the cement plant used the as-received prep-waste at 20% replacement of the regular plant fuel, and the composite fuel blend was used directly in the precalciner and rotary kiln as the primary fuel. The plant expressed genuine interest in considering the use of prep-waste after their current contract with the fuel supplier is expired. However, they would prefer a source closer to plant for economical reasons.

Although other cement plants have also expressed interest in the prep-waste use, their prior commitments to the fuel supplier(s) coupled with concern over economical shipment prevented the implementation of the technology during the time frame of the project. To lesser extent the reliability of source and consistency of material in terms of moisture, volatiles, and ash content, were also of concern.

Our meetings and the ensuing discussions with the cement plants and prep-waste suppliers also suggest the perception that the cement plants are entitled for an economic incentive for using the prep plants "waste," whereas the prep-waste suppliers contend that they are not getting their energy-laden material's worth, hence they would prefer to keep the material landfilled until they can realize the anticipated revenue from this material.

CTLGroup feels that the current non-conducive climate is most likely transitory. The cement plants have a genuine interest in this material, and, at an opportune time in future any combination of affordable shipping, consistency of prep-waste composition, and reliability of supply will result in its acceptability and commercialization at cement plants. Therefore, CTLGroup intends to keep the contacts with the cement plants and mine sites ongoing and forward looking. We also stressed with the prep plants the economic incentives from ICCI and DCEO for system modification(s) which can help improve processing and make the material economically attractive.

CTLGroup also believes that in order to expedite the commercialization of the prep-waste technology, the local and state governments could pass legislation to encourage the waste and by-product users with economical incentives. CTLGroup, ICCI, and DCEO may consider forming a consortium to pursue this avenue.

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ICCI Project Number: 04-1/4.1A-2
Principal Investigator: Javed I. Bhatti, CTLGroup
Other Investigators: John Gajda, and Donald Broton, CTLGroup
Project Manager: Francois Botha, ICCI

LIST OF EQUIPMENT PURCHASED

No equipment was purchased for this project.

PUBLICATIONS AND PRESENTATIONS REPORT
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