

FINAL TECHNICAL REPORT
September 1, 2002, through March 31, 2004

Project Title: **COAL-PREP-WASTES IN CEMENT MANUFACTURING – A
DEMONSTRATION PHASE**

ICCI Project Number: 02-1/3.1E-1
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ABSTRACT

This study entails the feasibility of demonstrating the use of Illinois coal prep-wastes in the manufacture of portland cement on a commercial scale. Due to the high fuel value, the coal prep-waste was used as a supplementary fuel at a commercial cement plant. The prep-waste was used at a 15% substitution of the plant coal during a 24-hour long demonstration. The plant, operating on semi-dry process with preheater/precalciner configuration, ran smoothly without experiencing any noticeable operational, kiln, emissions, or product variations. The plant experienced a 10-15% fuel saving. No abnormalities in the functioning of the precalciner, preheater, or the rotary kiln were observed. The clinker produced during the demonstration was similar to that produced before and after the demonstrations. The portland cement thus produced was in compliance with the ASTM C 150 specification.

The prep-waste used in the demonstration was procured from Marissa Mine of Randolph County in southern Illinois. The cement plant that participated in the demonstration was the Buzzi Unicem Cement plant (formerly Lonestar Industries, Inc.) in Greencastle, Indiana. Nearly 50 tons of prep-waste was dredged from the Marissa slurry pond. Since the material was wet (25-30% moisture) and finer than the typical coal, it was partially air-dried and mixed with waste coal to facilitate drying and to make it flowable prior to shipping to the cement plant.

Microscopical examination of the clinker produced during this demonstration confirmed the presence and distribution of major phases typical of a normal clinker. Similarly, the cement produced during the demonstration showed chemical and physical properties comparable with those of commercial cements when tested for compliance with ASTM C 150 specification.

The commercial-scale demonstration at the cement plant confirmed that the use of the coal prep-waste lead to smooth operation without any process, material, fuel, operational, environmental, or product variations. This approach should impart both material and environmental benefits to both the coal mines and cement plants that are willing to participate in the exploration of this technology. The Buzzi Unicem has already expressed interest in longer duration demonstration(s) at their cement plants in the Midwest region including the plants at Oglesby in Illinois, Cape Girardeau in Missouri, and of course at Greencastle in Indiana.

EXECUTIVE SUMMARY

The objective of this project was to use Illinois coal prep-wastes in portland cement manufacturing. Since prep-wastes are frequently high in fuel value, they were aimed to partially replace commonly used fuel (coal or coke) at a cement plant. The presence of high coal content (between 60-75%) in the prep-wastes was responsible for providing significant saving in purchased fuel requirements during the clinker firing at the cement plant.

Of the several prep-waste samples previously collected and tested for fuel values from different mining operations in Illinois, prep-waste from Marissa coal mine in Randolph County, southern Illinois, was used for the commercial demonstration at Buzzi Unicem Cement plant in Greencastle, Indiana. Nearly 50 tons of prep-waste was dredged from the slurry pond at the Marissa mine. The material was mixed with waste coal at the mine site and vigorously air-dried to make it flowable prior to shipping to the cement plant. About 80 tons of processed material was delivered to the cement plant for a 24-hour demonstration run.

The prep-waste samples from Marissa mine showed loss on ignition (L.O.I.) ranging roughly from 60-75% and the corresponding fuel values of 6000 to 7500 Btu/lb. The material was essentially a "low-grade" coal and prompted an investigation on its use as such. Of the several cement plants contacted for demonstration, Buzzi Unicem Cement plant in Greencastle, Indiana, was the most suitable because of its configuration and interest in the use of waste fuels; the plant is a semi-dry, single stage preheater/precalciner type and uses waste-derived fuels for its clinkering operation.

The cement plant used the prep-waste at 15% substitution of coal over a period of 24 hours. During the demonstration, several parameters including: precalciner fuel feed rate, ease of prep-waste conveying, fuel saving, emissions, clinker production and characterization, and cement production and properties were observed.

During the demonstration, the plant ran predictably smooth without any operational, emission, and product anomalies. The plant realized a fuel saving close to 15% with respect to precalciner operation. No abnormalities in precalciner and/or the rotary kiln thermal profile were noted, and there was no adverse impact on emissions release. The clinker was similar to that produced before and after the demonstration and the resulting cement was in compliance with ASTM C 150 specification.

The study suggests that the concept of using Illinois coal prep-waste as a partial fuel in cement manufacturing can lead to an efficient and high-volume utilization. This should impart several material, operational, energy, environmental, and economic benefits to coal mines that generate prep-wastes, as well as to the cement plants that are willing to use them in cement manufacturing. It may be mentioned that the Buzzi Unicem has already expressed interest in long-term demonstrations (7-10 day duration) at their cement plants in the Midwest region including the ones at Oglesby in Illinois, Cape Girardeau in Missouri, and of course, at Greencastle in Indiana.

OBJECTIVES

The objective of the project was to demonstrate the use of Illinois coal prep-waste (as a fuel supplement) for manufacturing portland cement. Being rich in heat value (exceeding 6000 Btu/lb), the prep-wastes could partially substitute for frequently used fuels such as coal or coke for the energy-intensive process of cement manufacturing.

Based on preliminary calculations and approximations, the following objectives seem plausible:

- A 15-20% substitution rate of prep-waste with regular fuel in the four cement plants in Illinois (combined capacity of 2.5 million tons/year), could consume 120,000 tons of waste each year to manufacture commercial cement.
- Depending upon the energy recovery from the prep-wastes, the cement plant using 15% prep-wastes with 65% loss on ignition should be able to save about 10% energy input.
- This "waste-to-energy" approach would generate a saleable product while significantly reducing wastes and related environmental stresses in Illinois.

Both the coal preparation facilities and cement plants should be able to benefit from this technology. Additional large-scale and longer duration demonstration(s) would, however, be useful to develop this technology and fully realize the benefits of consuming large volumes of prep-wastes in cement manufacturing.

INTRODUCTION AND BACKGROUND

More than 5 million tons of prep-wastes are generated from coal cleaning operations in the State of Illinois each year. These prep-wastes are routinely disposed in nearby landfills. Years of accumulation of these wastes presently have amounted to several hundred million tons where their components can potentially leach into the ground and cause environmental and ecological imbalance. Except for occasional use as mining backfill, these wastes have not found a beneficial reuse.

Prep-wastes typically consist of gangues and varying contents of residual coal that can realize significant fuel value. By virtue of the intrinsic coal content in the prep-waste, it is anticipated that the prep-wastes would facilitate fuel savings when used as a fuel supplement in the energy-intensive process of cement manufacturing. This would lead to multifold benefits of: 1) energy conservation, 2) waste recycling, and 3) reducing environmental stress in the locality.

Therefore, this study reports a commercial demonstration on the use of coal prep-waste as fuel supplement in cement manufacturing. A high L.O.I. prep-waste from Marissa mine in southern Illinois was used at Buzzi Unicem Cement plant in Greencastle, Indiana. Nearly 50 tons prep-waste was preprocessed prior to shipping to the cement plant for a 24-hour demonstration. Critical operational parameters were observed during the demonstration with the realization of several operational, material, energy,

environmental, and production benefits. The clinker and cement produced during the demonstration were evaluated in accordance with ASTM C 150 requirements.

EXPERIMENTAL PROCEDURES

The investigational steps that lead to the commercial demonstration were as follows:

Task 1 – Material Procurement

Several samples of prep-wastes were collected from Illinois coal-processing facilities identified as Rend Lake, Monterey, White County, Randolph, and Turriss mines. The prep-waste from Randolph County’s coal mine in Marissa, because of its high fuel value (based on high loss on ignition, L.O.I.*), was selected for further evaluation as a fuel supplement in cement manufacturing.

Task 2. Material Characterization

Using X-ray fluorescence (XRF), the prep-wastes samples were analyzed for their chemical composition of SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, SO₃, Na₂O, K₂O, and L.O.I. Selected prep-waste(s) with high L.O.I. were also subjected to differential scanning calorimetry (DSC) to determine the heating value and the temperature of volatilization of any combustible organics that might be present; the prep-waste(s) were also tested for short proximate analyses and trace metal levels.

RESULTS AND DISCUSSION

Based on the oxide composition and the fuel values of the prep-wastes tested above (as shown in Table 1 and Figure 1), the prep-waste from Randolph mine in Marissa (slurry #2 area) was selected for further evaluation.

Table 1. Composition of prep-wastes from Illinois coal mines, wt. %

Analyte	Monterey	Turriss	Rend Lake	Randolph slurry #1	Randolph slurry #2
SiO ₂	27.38	43.24	46.33	22.55	29.70
Al ₂ O ₃	9.82	11.17	13.76	6.80	8.35
Fe ₂ O ₃	12.63	9.6	3.79	12.79	3.14
CaO	17.0	3.8	0.63	12.32	2.31
MgO	0.67	1.13	1.06	0.53	0.79
SO ₃	5.26	3.25	0.32	11.11	1.33
Na ₂ O	0.4	0.74	2.29	0.18	0.49
K ₂ O	1.18	2.04	0.73	0.92	1.29
L.O.I.	24.5	23.94	30.27	31.80	52.69

The data indicate that the prep-waste from Randolph mine in Marissa (slurry #2) is high in L.O.I. suggesting the material to have a reasonable fuel content. The material is also low in sulfur, which is always preferred in cement operations to avoid by-pass plugging.

* L.O.I. is also an indication of the carbon content in prep-wastes.

In general, the prep-wastes are also rich in SiO_2 , Al_2O_3 , Fe_2O_3 , and CaO , suggesting that the ash left after combustion would be beneficially incorporated into the cement clinker. The material is fine in nature; typical particle size distribution is shown in Table 2.

Table 2. Particle Size Distribution of Randolph Slurry #2, wt. %

Sieve size	# 5	# 20	# 50	# 100	Passing # 100
Retained	0.7	5.2	19.3	22.1	52.7
Cumulative	0.7	5.9	25.2	47.3	

The DSC plot of the prep-waste from Randolph slurry #2 evaluate the heat content and characteristics of the volatile organic matters present therein, is shown in Figure 1.

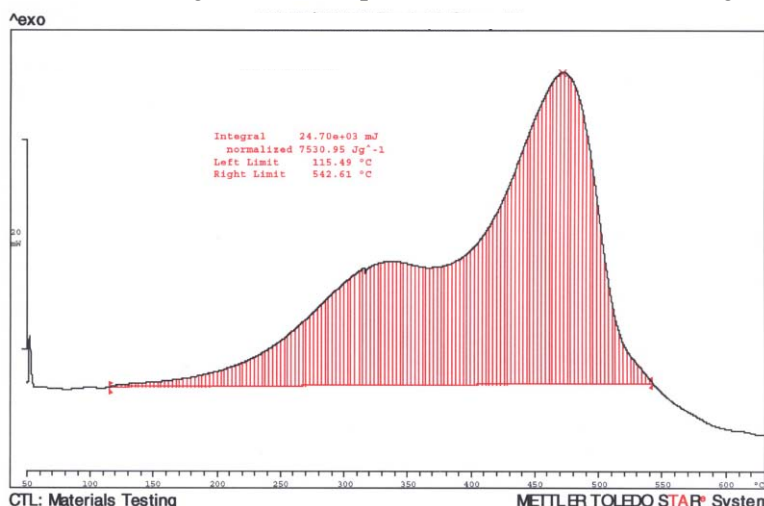


Figure 1. DSC plot showing exothermic peaks in Randolph slurry #2 prep-waste

Large exothermic peaks starting around 200°C and peaking 470°C indicate a heat release by combustion of hydrocarbons from residual coal in the prep-wastes. However, the exothermic hump at lower temperatures is indicative of the presence of volatile and combustible components in the wastes. This latter material may be released at temperatures so low that it may escape combustion in the cement kiln as unburned volatile organic compounds (VOC's) and cause emission problems.

The proximate analysis of 2 prep-waste samples taken from Randolph slurry #2 location is shown in Table 3. The data indicate that the prep-waste, with an average fuel value of nearly 6775 Btu/lb, is more like a “low grade” coal that can be used as a fuel supplement in a cement manufacturing – and are therefore selected for the demonstration.

Table 3. Proximate analyses of Randolph coal prep-waste samples, wt. %

Proximate analysis	Sample 1	Sample 2	Average
Moisture	4.71	3.91	4.71
Ash	31.94	42.37	37.16
Volatiles	28.61	25.97	27.29
Fixed Carbon	34.74	27.71	31.23
Btu/lb	7522	6028	6775
Sulfur	2.34	2.53	2.43

Trace metals analysis of a composite prep-waste sample is shown in Table 4. The level of trace metals in the prep-waste is too low to be a cause of concern.

Table 4. Trace metals in coal prep-waste

Metals	Concentration, $\mu\text{g/g}$
Arsenic	22
Barium	194
Cadmium	1
Chromium	57
Lead	6
Mercury	0.06
Selenium	<1
Silver	3

Task 3. Raw Feed Formulation

Since the prep-waste was to be used as a fuel supplement in cement manufacture at a low substitution rate (15% by weight of coal), the ash resulting from combustion would be too small to impact the kiln feed formulation, hence this task was not considered necessary.

Task 4. Identifying the Method of Prep-Waste Introduction

As is mentioned before, the prep-waste from the Randolph mine in Marissa, being rich in fuel value (6780 Btu/lb), low in sulfur (2.44%), and having no trace metal issues, was actively considered for use as a partial fuel in cement manufacturing. Cement plants with preheater/precalciner configurations were approached to conduct a commercial-scale demonstration. Amongst these plants, Buzzi Unicem plant in Greencastle, Indiana agreed to participate.

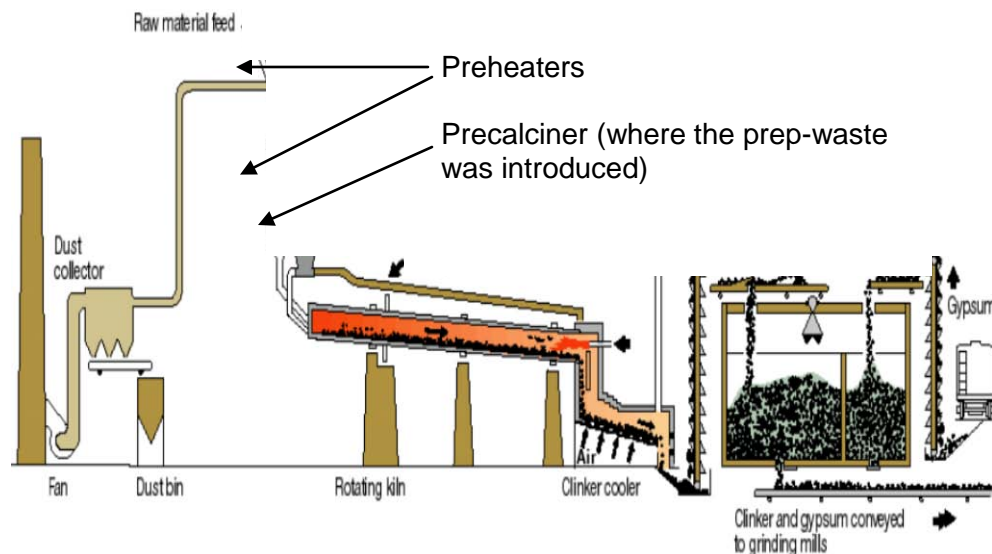


Figure 4. A cement plant showing preheater/precalciner configuration - coal prep-waste was introduced in the precalciner as fuel supplement during the demonstration

The Greencastle cement plant is a semi-dry process of preheater/precalciner configuration with an annual clinker production capacity of over 1.5 million tons; the plant consumes about 700,000 tons of coal annually. The rotary kiln operates principally on waste derived fuels. The prep-waste was introduced as a partial fuel substitute in the precalciner. A typical preheater/precalciner cement plant is shown in Figure 4. Greencastle cement plant planned a 24-hour trial burn with the prep-waste using 15% substitution for coal in the precalciner. This required about 80 tons of dried prep-waste to be delivered to the plant.

Task 5. Prep-Waste Preparation

Surveying and Sampling of Slurry #2 Pond. Prior to the dredging of the prep-waste from the Randolph mine in Marissa, a survey of the slurry #2 pond was carried out and several samples were collected (Figure 5), for a preliminary estimation of dryness (based on moisture content), and fuel value (based on L.O.I.). The samples were collected from the top 6-9 inches of the pond surface.

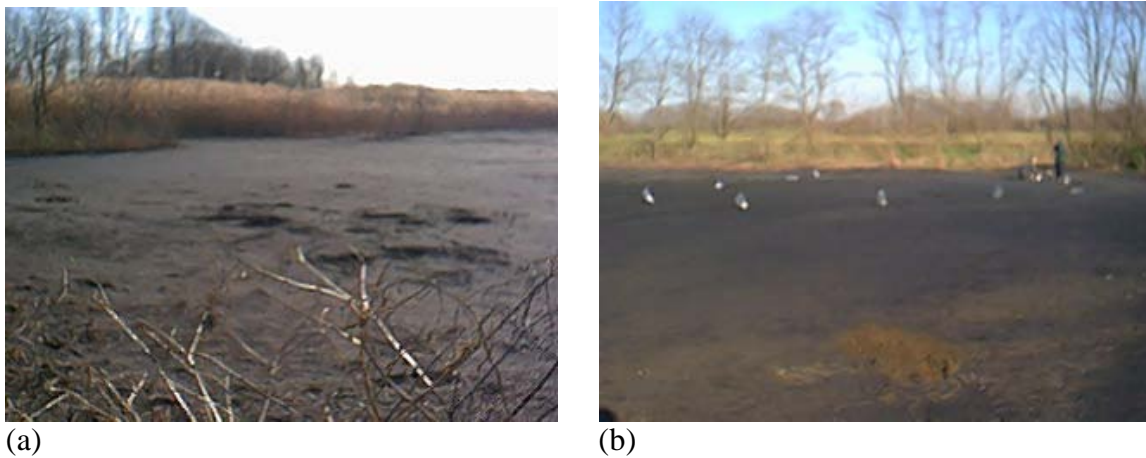


Figure 5. Slurry #2 pond of Randolph mine in Marissa (a); and surveying and sampling locations (b) of the prep-waste

Top section of the slurry was generally drier than the overall material in the pond. The data on moisture and L.O.I. is shown in Table 5.

Table 5. Moisture and L.O.I. in prep-waste samples, wt. %

Sample #	1	2	3	4	5	6	7	8	9	10	11	12
Moisture	27.3	29.5	30.4	26.7	27.8	28.7	26.0	27.7	27.3	25.6	29.7	26.2
L.O.I.*	64	66	76	65	65	66	74	63	60	61	57	71

L.O.I. was determined at 950°C on dry basis

The moisture content of the prep-waste ranged from 25.6 to 30.4% with an average of 27.7%. The L.O.I. of the samples ranged from 57 to 76% with an average of 66%. The L.O.I. suggested that although the material had a reasonable fuel value, it had to be dried close to 10% moisture in order for it to be flowable and blendable with the regular coal at Greencastle plant for the demonstration.

Dredging and Hauling of Prep-Waste. Based on the data in Table 4, it appeared that the prep-waste was reasonably uniform in moisture as well as L.O.I. A large amount of material was collected by scraping the top 8-12 inches of the slurry over an approximately 1000-ft² area of the pond (Figure 6). In order to have 50 tons of dry material, several trucks of the prep-waste were hauled and delivered to an open staging (drying) site.



Figure 6. Dredging of prep-waste from slurry #2 pond

Drying and Preprocessing of Prep-Waste. The material was wet and sticky (moisture close to 30% and finer than typical coal). In order to dry it without elevated heating (to avoid the release of emission-related species), the material was spread out in the open for air-drying (Figure 7). The material was frequently turned to expedite drying and as far as possible kept under cover to protect from rain or snow. In order to further enhance the aeration and flowability of the material, a dry coal fraction available at the mine site was blended with the prep-waste to have a bulk amount of about 80 tons.



Figure 7. Coal prep-waste being spread out at drying site

The final material was tested for proximate analysis for fuel value and sulfur (see Table 6). As can be seen, the blending of the prep-waste with coal fraction improved the fuel value from 6775 to 9697 Btu/lb whereas the sulfur content changed only marginally. The moisture content of the final material was about 10% by weight; the material was acceptably dry and flowable.

Table 6. Proximate analysis of prep-waste blend, wt. %

Proximate analysis	Prep-waste blend
Moisture	2.00
Ash	21.66
Volatiles	32.91
Fixed Carbon	43.43
Btu/lb	9697
Sulfur	2.67

Prep-Waste Delivery to Cement Plant. The cement plant required the material to be non-sticky and flowing so that it could be easily introduced to the system via screw conveyor. Once the material was dry and flowable to the cement plant requirements (moisture level 10% by wt.), nearly 80 tons (3 truck loads) of the material were delivered to Greencastle cement plant in Indiana (Figures 8). The material was unloaded in their covered coal storage area (Figure 9), where it could be blended with the plant coal* and conveyed to the coal mill for its eventual use as fuel supplement. Prior to movement to the conveyor system, the material was blended with the plant coal using a front-end loader at approximately 6 coal to 1 prep-waste ratio (i.e. 15% prep-waste substitution).



a) Prep-waste being loaded on trucks



b) Trucks leaving for cement plant

Figure 8. Prep-waste loading and delivery to Greencastle cement plant

* Plant coal is the regular coal used as precalciner fuel



Figure 9. Prep-waste trucks being unloaded in covered storage at Greencastle plant

Task 6. Kiln Burn and Operational Observations

Blending and Pre-Grinding. The demonstration began on March 16, 2004, at approximately 1 pm when the prep-waste was introduced to the kiln system of the Greencastle Cement plant (Figures 10 and 11). The plant is a semi-dry process with a precalciner and a single-stage preheater. The plant primarily uses waste-derived fuel for its rotary kiln, and coal is the primary fuel for precalciner.



Figure 10. Greencastle Cement plant showing precalciner/preheater tower



a) Precalciner



b) Single-stage preheater



(c) Rotary kiln

Figure 11. Precalciner (a) preheater (b) and rotary kiln (c) of Greencastle Cement plant

As mentioned earlier, the prep-waste was blended with the plant coal initially at 10% level; the blend rate was increased to 15% as the operation progressed. The mixture of prep-waste and plant coal had a heat value of 12,054 Btu/lb and sulfur level of 1.54%. A short proximate analysis of the mixture is shown in Table 7.

Table 7. Short proximate analysis of prep-waste (15%) – plant coal mixture, % wt.

Analyses	As-received	Dry basis
Moisture	5.9	–
Ash	10.06	10.69
Btu/lb	12054	12810
Sulfur	1.54	1.64

The mixture traveled to the coal mill via screw conveyor where it was ground and introduced to the precalciner. A pile of blended mixture of prep-waste and coal being conveyed to coal mill is shown in Figure 12. Even though this material was finer than typical delivered coal, the mixture required grinding to be useable. Due to its inherent moisture content, some operational adjustments had to be made to accommodate this material. Slightly more energy was used to grind the material before introduction into the burning system.

**Figure 12. Piles of prep-waste and plant coal blends being conveyed to coal mill**

Operational Parameters Monitored. As the material entered the system, it became clear that tracking this one small component would be difficult. The 15% substitution of the precalciner coal is only a small portion of the energy needed to complete the formation of cement clinker. The control room constantly monitored the conditions within the kiln system and made changes as necessary and a multitude of other parameters to keep the conditions within the normal operating conditions. The parameters observed are shown in Table 8.

Table 8. Operational parameters observed during prep-waste demonstration

Calciner coal blower rate (a function of ease or difficulty in conveying)	Coal feed rate to precalciner	Preheater exit CO	SOx level
Cyclone cone draft (air-flow rate)	Fly ash rate	Preheater exit O ₂	Stack opacity
Fuel flow to coal mill	Kiln feed rate	NOx level	

It must be emphasized that the tracking of a single, small component is difficult as normal changes in the actual plant coal and other operational parameters may also cause operational changes, which the pre-waste may not have contributed to. The observations made on the above parameters are categorized and summarized in Table 9.

Table 9. Categorized summary of observations made during demonstration

Categories and operational parameters	Summary of observations and comments
Material	
Calciner coal blower rate (a function of ease or difficulty in conveying)	Marginally higher precalciner blower AMPS indicating the material a little harder to convey – most likely because of the inherent moisture, a drier material will be much easier to convey
Cyclone cone draft (air-flow rate)	The by-pass showed no change in flow rates, draft pressure was normal – no adverse plugging observed
Fuel	
Fuel flow to coal mill	A little more dry coal needed in coal mill – as expected a little more coal used to adjust the heat level
Coal feed rate to precalciner	Calculated coal feed rate was little more than normal – again coal adjustment needed in precalciner to maintain fuel level
Production	
Fly ash rate	No fly ash required to adjust the kiln feed composition – ash from prep-waste adequately incorporated in the kiln feed
Kiln feed rate	Normal kiln production – though a slight increase in kiln production was observed towards later stages of the demonstration
Emissions	
Preheater exit CO	No change observed
Preheater exit O ₂	No change observed
NOx level	No change observed
SOx level	No change observed
Environmental	
Stack opacity	No adverse effects

As can be seen from the data in Table 8 on the operational observations made during the demonstration, the use of prep-waste rendered a beneficial contribution as a fuel supplement and the overall operation, as expected, was smooth and predictable. There were no adverse observations on material, fuel, production, emissions, or environmental issues during the demonstration. Slight improvement in kiln production was observed towards the later stages of demonstration.

The test burn was completed at approximately 9 am on March 17th 2004. Samples of clinker and cement were obtained from the plant for testing at approximately 12 hours prior to test burn, after 12 hours of prep-waste usage, and 12 hours post burning. The material was shipped to CTL for examination. The cements were subjected to ASTM C 150 chemical and physical testing.

Task 7. Characterization and Evaluation of Clinkers

The clinkers produced during the demonstration were characterized for their physical and chemical properties and compared with those produced before and after the demonstration. The following qualitative and quantitative tests were employed.

X-Ray Diffraction (XRD). Ground clinker samples were subjected to XRD analyses to identify major crystalline phases and any variations caused by prep-waste use in the precalciner. It must be emphasized that a 15% use of prep-waste should not incorporate sufficient ash to the kiln feed to cause any variation in the clinker phases. XRD plots of clinker produced during the demonstration and the ones produced before and after are shown in Figure 13.

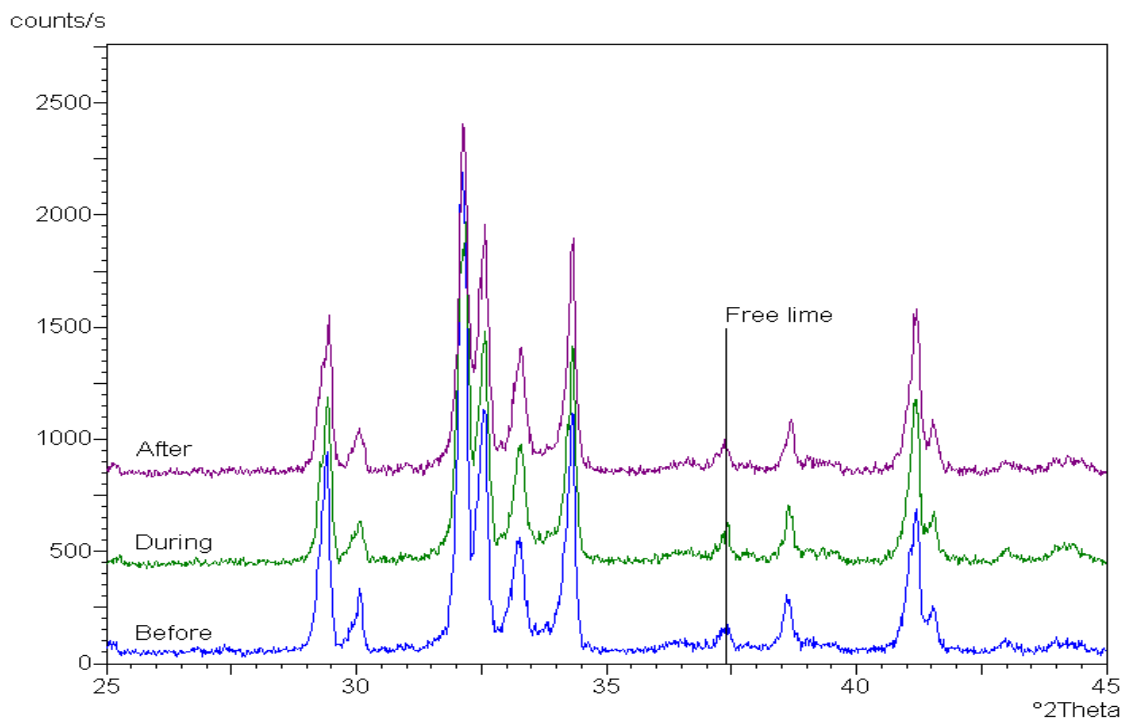


Figure 13. XRD pattern of clinkers show major C_3S , C_2S , C_3A , C_4AF free-lime peaks

In Figure 13, alite (C_3S) and belite (C_2S) phases are indicated by peaks at 32.2, 32.7, and 34.4 degrees. Peaks for tricalcium aluminate (C_3A) and tetracalcium aluminoferrite (C_4AF) are at 33.3 and 34.1 degrees respectively; peaks for C_4AF are appearing as a shoulder in these XRD patterns. A peak at 37.4 degrees indicates the presence of free lime (CaO).

The identical XRD patterns of clinkers – showing appropriate distribution of major clinker phases and only traces of free lime content – suggests no adverse effects during the demonstration as compared to the control clinkers produced before and after.

X-Ray Fluorescence (XRF). The oxide analysis by XRF and the computed Bogue compounds for clinkers are shown in Table 10. The data also confirmed the presence of major phase in the clinkers.

Table 10. Chemical composition* and Bogue analyses of clinkers, wt. %

Analyte	During	Before	After
SiO ₂	21.19	20.77	21.17
Al ₂ O ₃	5.81	5.78	5.95
Fe ₂ O ₃	2.31	2.42	2.36
CaO	67.62	68.01	66.95
MgO	0.99	0.85	1.00
SO ₃	1.15	1.16	1.19
Na ₂ O	0.13	0.10	0.12
K ₂ O	0.74	0.77	0.80
Bogue Compounds			
C ₃ S	68	73	65
C ₂ S	9	4	12
C ₃ A	11	11	12
C ₄ AF	7	7	7

* Analyses on ignited basis

Again, as can be seen, there are similarities between the contents of major phase for the demonstration as well as the control clinkers – confirming that there was no adverse effect on clinker chemistry as a result of prep-waste use in the precalciner. The chemical composition and the computed Bogue compounds in clinkers show them to be of Type I/II.

Optical Microscopy. Polished sections of clinkers were examined by microscopy to determine relative quantities and distributions of the major phases. Photomicrographs in Figure 14 and 15 show a reasonable distribution of the major phases in all clinkers. The field length (FL) of the photomicrographs is 560 μ m.

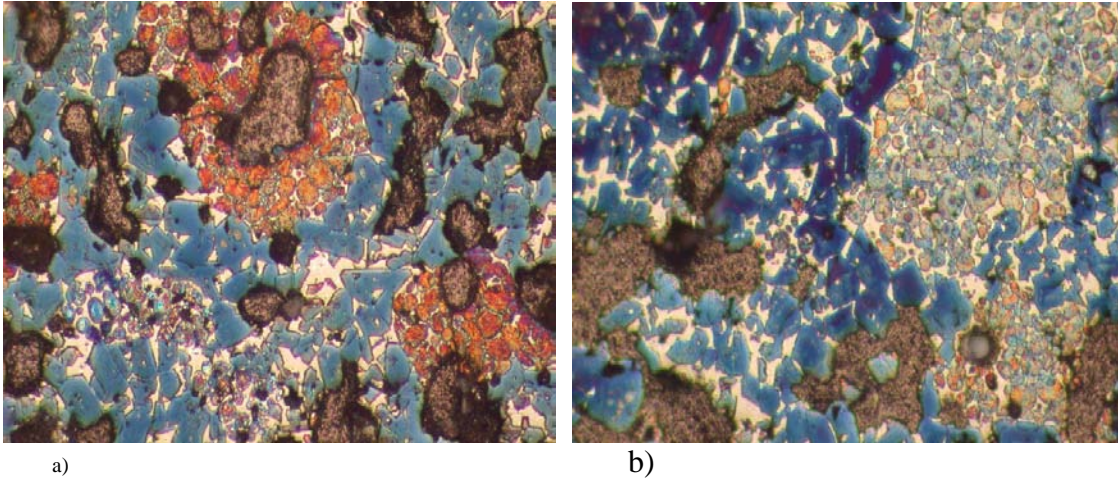


Figure 14. Major phase distribution in clinkers before (a) and after (b) demonstration (field length = 560 μm)

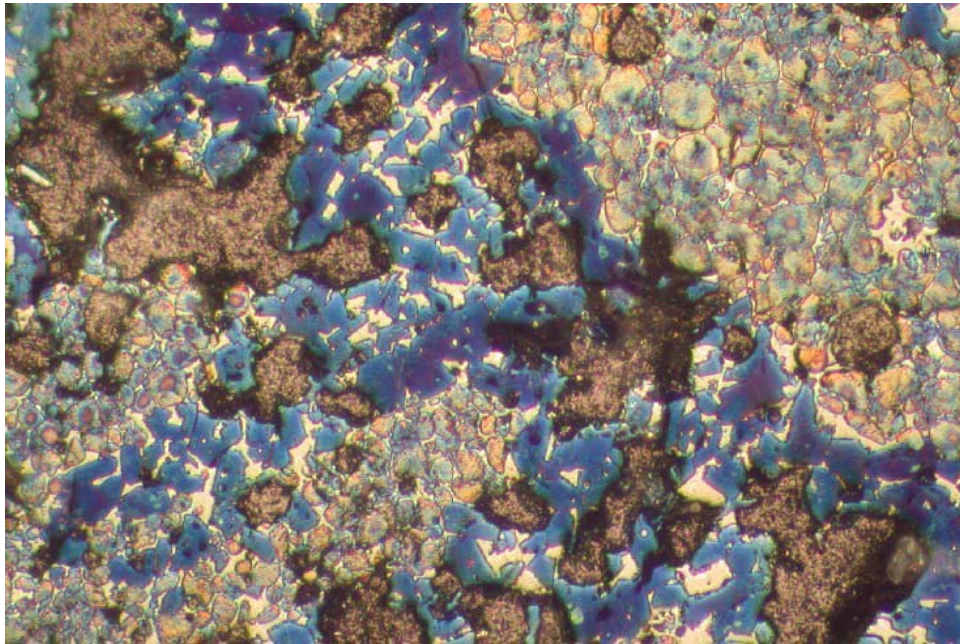


Figure 15. Clinker produced during demonstration show formation and distribution of major phases, (field length = 560 μm)

Large angular (bluish) crystals in the micrographs are C_3S (alite), the round (brownish) crystals with lamellae are C_2S (belite); the interstices are composed of C_3A (tricalcium aluminate) and (C_4AF) tetracalcium aluminoferrite – these are also known as the melt phases. Large random round areas are pores. Higher porosity can lead to better grindability of clinkers.

As can be seen from the micrographs, the distribution of alite, belite, and the interstitial phase are typical for all three clinkers. This clearly suggests that the clinker produced

during the demonstration is similar in its mineralogical makeup to those produced before and after the demonstration.

Task 8. Production of Cements

The cements produced from clinkers before, during, and after the demonstration were procured from the plant for testing and evaluation as follows:

Task 9. Testing of Cements per ASTM C 150 Specifications

The cements were tested for compliance with ASTM C 150, "Standard Specification for Portland Cement." The test regimen includes both chemical and physical tests on compressive strength, air content, time of set (both initial and final), and early stiffening.

Chemical compositions and Bogue analyses of the blended cements as determined by XRF are shown in Table 11. The standard requirements for Type I/II cement are also given for comparison.

Table 11. Chemical composition* and Bogue analyses of cements, wt. %

Analyte	During	Before	After	Standard requirements
SiO ₂	20.69	20.91	20.84	20 (min)
Al ₂ O ₃	5.73	5.69	5.93	6 (max)
Fe ₂ O ₃	2.28	2.26	2.25	6 (max)
CaO	65.69	65.49	65.17	N.A.
MgO	1.24	1.31	1.41	6 (max)
SO ₃	3.35**	3.31	3.28	3 (max)
Na ₂ O	0.14	0.13	0.14	N.A.
K ₂ O	0.77	0.80	0.80	N.A.
Bogue Compounds				
C ₃ S	59	57	54	N.A.
C ₂ S	15	17	19	N.A.
C ₃ A	11	11	12	N.A.
C ₄ AF	7	7	7	N.A.

*Ignited basis, ** cements exceeding 3% sulfate are routinely checked by plant to meet ASTM C 1038

Chemical data indicate that cement produced during the demonstration is comparable to those produced normally and meets the standard requirements for Type I/II cement. It may be mentioned that cement exceeding 3% because of the presence of calcium sulfate is routinely tested for compliance with ASTM C-1038 "Standard Test Method for Expansion of Portland Cement Mortar Bars Stored in Water." The amount of expansion in the mortar bar is related to the amount of calcium sulfate present in the cement. Excessive expansion occurs when the cement contains too much calcium sulfate.

The cements were subjected to a series of physical tests as required by the ASTM C150 specification. They included fineness, compressive strength, autoclave expansion, air content, time of set (initial), and early stiffening. Test data are shown in Table 12; standard requirements for each test are also given for comparison.

Table 12. Data on ASTM C 150 tests on cements

	During	Before	After	Requirement for Type I/II cement
ASTM C 204 - Fineness, air permeability (Blaine), m²/kg				
	346	337	340	280 (min)
ASTM C 109 - Compressive strength, psi				
1-day	2190	2000	2080	Optional
3-day	3650	3560	3810	1740 (min)
7-day	4880	4390	4390	2470 (min)
28-day	5700	5420	5950	4060 (min)
ASTM C 151 – Autoclave Expansion				
	0.05	0.03	0.05	0.8 (max)
ASTM C 185 – Air Content				
	6.9	7.2	7.8	12 (max)
ASTM C 191 - Time of set, minutes				
Initial	60	75	60	45 (min)
ASTM C 451 - Paste false set - early stiffening				
	83	76	74	50% (min)

The data show that the demonstration cement meets the physical tests requirements of the ASTM C 150 specification, as did the cements produced before and after the demonstration. The cements show normal setting and strength development characteristics within the acceptable range for Type I/II cement. It might be noted that the 7-day compressive strength of the demonstration cement significantly exceeds those of the normally produced cements.

It may be pointed out that as a result of successful demonstration at the Greencastle Cement plant, and the promising operational indicators realized, the Buzzi Unicem U.S.A. has already expressed interest in long-term demonstration(s) on the use of coal prep-wastes at their cement plants in the Midwest– including the plants at Oglesby in Illinois, Cape Girardeau in Missouri, and of course at Greencastle in Indiana.

CONCLUSIONS AND RECOMMENDATIONS

Coal prep-wastes appear to be a viable fuel supplement. By virtue of being rich in fuel value, they can be used as fuel by partially replacing coal in cement manufacturing.

Given the conservative approach exercised by the cement plant, demonstration showed that the prep-waste could replace in excess of 15% coal. This could translate to several interrelated benefits such as: 1) high volume recycling of the prep-waste, 2) eliminating waste accumulation, 3) reducing the local environmental stress, and at the same time 4) conserving the natural resources that are otherwise purchased.

During the use of prep-waste, the cement plant demonstrated a predictable and smooth run – the plant did not encounter any significant operational, processing, material, emission, product, and environmental problems.

The clinkers produced during the demonstration exhibited similar chemical and mineralogical properties to those produced before and after the demonstration. Likewise, cements produced during the demonstration also exhibited similar chemical and physical properties conforming to the general purpose Type I/II cement.

Overall, as demonstrated by the large-scale commercial run, the concept of using prep-waste as partial fuel in cement manufacturing, will positively address the issues of waste management and the related environmental stresses faced by the coal mines and cement plants in the region. Both industries would benefit economically as well as environmentally in adopting this technology. A full-scale demonstration is recommended to assist in demonstrating these benefits. In this regard, based on our experience, observations, and dealing with both the coal mines and cement plants – additional points need to be considered are as follows:

- 1) A survey of the mine sites revealed significant differences with respect to physical properties of the coal waste ponds (slurry are generally rich in fuel value but very high in moisture contents). Cement plants require uniformity in composition or compositional analysis to determine potential variability in materials supplied. Compositional mapping of the areas (with respect to, say, proximate analyses) is imperative to fully utilize the material. Actual depths of sampling/testing need to be determined once cores are obtained and examined petrographically.
- 2) Modes of introduction of prep-waste into the cement kiln system should be investigated. The present commercial run was accomplished on semi-dry material. This was necessary to utilize the material in the precalciner. Other modes of introduction into cement kiln burning system may be equally beneficial. Depending on the location of plant and kiln system, a different mode of prep-waste introduction may be necessary and trial burns will be required for each type of kiln system (for instance, mid-kiln injection and insufflation from the firing end are other options).

- 3) Transportation costs are significantly higher on wet material. Moisture of the original material dug from ponds was over 30%. Methods of drying the material prior to shipment are worth investigating.
- 4) Two-way transport (back hauling) may reduce cost for shipping. If the trucking company can obtain full loads of material to bring back to the direction of the prep-waste costs may be reduced.
- 5) The nearly 80 tons of material used for production showed no changes in the clinker or cement composition due to its inorganic constituent contribution. To accurately assess the feasibility of large amounts of this material, a longer, possibly a week long run could yield significantly more information. Test burns for 7 days would be most beneficial using different kiln systems. Each kiln system would have time to stabilize and different feed rates could be used to determine maximum utilization of the prep-waste.

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