

Utilization of Whey/Lactose as an Industrial Binder

Arthur Ferretti* and James V. Chambers

The emphasis of this work centers on the use of whey/lactose as the key ingredient of a binder system utilized in a patented industrial process having broad applications as an energy conservative resource recovery system. This work describes the use of whey/lactose in the manufacture of iron ore pellets and iron/steel pellets produced from iron fines captured in pollution control equipment. Commercial introduction of this process should effect large savings in the amount of energy expended in the making of iron ore pellets, permit recycling of iron/steel dust currently discarded and create a positive use for a currently desirable byproduct of the dairy industry. This process takes advantage of some chemical-physical properties of the extrusion blend to form materials of a structural strength which allows iron resource recovery. The whey/lactose is thought to improve the binding strength of the pellet/briquette and assist the ionic bonding structures which are critical to the ultimate strength of the recovered iron material.

Thrust of this paper will focus on the use of whey/lactose as an important ingredient in a binder system utilized in a patented extrusion process. This extrusion process has broad applications as an energy conservative resource recovery system. The initial patent (U.S. No. 3 567 811) which was issued March 2, 1971, dealt with a "method of producing strong fired compacts from iron or iron oxide containing material". The 1971 patent was followed by another patent application dealing with the processing of "bloated fly ash aggregates" (U.S. No. 3 765 920: October 16, 1973). The third patent (U.S. No. 3 857 715) was issued December 31, 1974, which described a process for making "extrusile hydraulic cement compositions containing alkali and carbohydrate". Since then, more applications have been identified using the extrusion process which have additional energy conservation and resource recovery implications.

In these patented processes, carbohydrates (as a class) have played an important role in the binder system used. The early developmental work utilized molasses as the primary carbohydrate of choice. However, later experimentation with whey/lactose demonstrated an improvement in the binding property of the binder over materials such as molasses and lignin. With whey/lactose in abundant supply, feasibility for its use in the extrusion process is possible.

Historically, the utilization of dairy wastes, namely whey and lactose, has been directed toward animal and human nutritional applications. Even though this type of carbohydrate-containing material was used as a binder in brick molding and as very durable buttermilk paint in Colonial America, its use as a modern industrial material has largely been overlooked.

The efforts reported herein describe work directed toward solving the mounting problems associated with the increasing amounts of dairy wastes that can no longer be utilized by traditional markets or disposed of in an environmentally acceptable manner. Broad industrial acceptance of this technology should not only relieve an overburdened environment but provide an economically attractive market for a substantial amount of these by-products.

Indianapolis Center for Advanced Research, Inc., "An Affiliate of Indiana University, Purdue University at Indianapolis", Indianapolis, Indiana 46202 (A.F.) and the Department of Animal Sciences, Purdue University, West Lafayette, Indiana 47907 (J.V.C).

EXPERIMENTAL SECTION

Apparatus. (1) Muller type batch mixer; (2) experimental extruder with vacuum accessory, Fate, Roote, Heath Co.; (3) drying oven or furnace (350 °C); (4) kiln (500+ °C).

Reagents. Carbohydrate: whey/lactose at 25% as a lactose solution, commercial grade. Mineral acid: hydrochloric acid at 37% solution, commercial grade. Wetting agent: derived as a condensation product of sorbose and polyethylene oxide, commercially known as "Liqui-Lass". Alkali: ammonium hydroxide at 26% solution, commercial grade.

Procedure. The following formulation yielded excellent results in terms of product density and hardness: 23 kg of pulverized magnetite ore (Cleveland Cliffs Mine), 375 mL of ammonium hydroxide, 65 mL of Liqui-Lass Surfactant, 340 mL of hydrochloric acid, 1200 mL of non-hygroscopic lactose, 1000 mL of water.

The above ingredients after being thoroughly mixed were extruded in a 25 mm column, cut to 40 mm lengths, and cured at 300 °C for 3 h in a circulating air oven.

The processes described encompass several applications, such as: the agglomeration of iron ore and iron dust (fines); the production of fuel briquettes from a mixture of refuse coal dust and sewage sludge; the production from fly ash of light weight aggregate for concrete; the extrusion of high strength, thin-walled cement pipe; the agglomeration of additives for steel alloying and others. Patents have been issued on several of these; others are being prosecuted. All have two elements in common: they are extrusion processes and utilize whey or lactose as a principal ingredient of the binder system.

RESULTS AND DISCUSSION

The results given below were obtained at a commercial laboratory from tests performed on samples of pelletized iron ore and were run in conformance with applicable ASTM standards.

Of particular importance are the reducibility index (38), indicating an ease of reduction to iron and the high crush strength and abrasion resistance.

Tumble Index: 1% through -28 mesh. Very good abrasion resistance.

Reducibility: Reducibility index 38, i.e., it will require 38 min to reduce 1 lb of pellets to 90% reduction in 3:1, H₂:CO at 916 °C in a set flow of reducing gas. This compares favorably with other commercial pellets under the same reducing conditions.

Hot Strength: Pellets fired at 1288 °C required 867 lb crushing load to produce failure. Pellets fired at 1260 °C required 406 lb. In this test 250 lb is considered acceptable. The test involves loading a pellet to failure in an atmosphere of CO₂:CO, 3:1, at 816 °C.

Similar results were achieved utilizing BOF (Basic Oxygen Furnace) dust which has an original zinc content of 3.6%. After curing at 1260 °C the zinc content was reduced to less than 2.5%, a decided advantage in the reutilization of this material.

In general a range of desired properties, specifically, hardness, strength, and abrasion resistance, can be induced in the end product by variation in the drying/curing cycle. This is desirable in some products such as cement pipe but of lesser importance in fly ash aggregates and fuel briquettes.

Iron Ore Pelletizing. With the rapid depletion of the higher grades of iron ore, it became necessary and economical in the 1950's to process ores containing larger proportions of impurities. To concentrate the iron, it is necessary to crush and grind the ores, separate the iron from the impurities, and reconstitute the iron ore into lumps, sinters, or pellets. This reconstitution is necessary for two reasons: (1) to provide a convenient form for shipment by rail or boat to the mills, and (2) to provide sufficient structural strength and integrity to survive the compressive loads and high gas pressures, temperatures, and flow rates encountered in a blast furnace without premature crumbling of the pellet. Prevention of premature crumbling is most important, for if the ore pellets deteriorate before entering the melting zone (temperature 1000–1500 °C), they will block or channel the gas flow and/or be blown out the top of the blast furnace. Therefore, great care is exercised at the iron ore processing plant to provide strong, high-quality pellets or sinters.

Pelletizing has, in the last decade, overtaken sintering as the preferred method of agglomerating iron ores.

Current pelletizing consists basically of two steps: (1) mixing of the ground ore with carefully controlled amounts of a binder (such as bentonite clay) and water and balling them on a large rotating disc or a balling drum, and (2) heating them in a kiln or grate furnace to about 1300 °C to fuse or indurate them into hard pellets.

In 1976, 250 million metric tons of ore was processed in North America, the largest proportion of which was prepared by pelletizing, as opposed to sintering. If manufactured by this new process, approximately 10 million tons of whey/lactose would have been required.

Recycled Iron Fines. The recycling of iron fines offers another potential use for the extrusion process described herein. The significance of recycling iron fines is succinctly stated by Pehlke (Pehlke, 1976). He states that for every ton of finished steel produced there is one-tenth of a ton of solid waste. Pehlke contends that this solid waste offers a valuable stockpile of raw materials in every steel mill. However, "the catch" he states "is that this material is in the form of dust or fine particles, and present technology allows only a portion of it to be reclaimed".

Extrusion Process. As described in the patents (Humphrey, 1971, 1973, 1974), the processes are straightforward and utilize equipment currently available from industrial suppliers. The key elements of the process are the unique constituents of the binder system and their proportions. The specific combination of the basic material with a carbohydrate (lactose), an acid, an hydroxide, and a surfactant (wetting agent) in the prescribed manner produces an extruded pellet (either a cylindrical rod or spherical ball) which has controllable size, porosity, density,

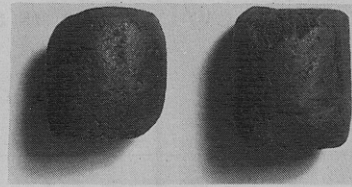


Figure 1. Iron ore fines, 600 °F.



Figure 2. BOF dust and iron ore, 600 °F.

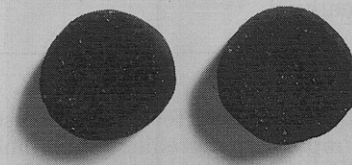


Figure 3. Steel Fines and mill scale, 1200 °F.

and compressive strength—in both the "green" (as extruded) condition or after firing. This firing, incidentally, can be effected at 260–315 °C, a considerable reduction in required energy over the current practice in the iron ore industry of firing at 1300+ °C (some materials, such as fluorspar, cure at room temperature). A ton of iron ore pellets produced by current methodology requires 600 000 BTU's compared to about 350 000 BTU's when a carbohydrate binder is used.

The basic process can be modified to handle a wide range of raw materials such as: (1) Iron ore fines of many species (taconite, magnetite, hematite, etc.) (Figure 1). (2) Dust from blast furnaces, open hearths, basic oxygen furnaces, and electric arc furnaces. This dust can be taken directly from the dust collector hoppers or the wet scrubbers. In the case of wet slurries, the moisture has to be lowered to about 18% by weight. This can be accomplished by blending with dry dust, thickening, or vacuum filtration (Figure 2). (3) Mill scale and steel fines over a wide range of particle sizes (Figure 3). (4) Miscellaneous steel waste from secondary processing. (5) Many other finely divided materials, such as coal, coke breeze and lime—all steel making additives.

The selected carbohydrate material utilized by Humphrey in most of his early development work was blackstrap industrial molasses. In recent work molasses was compared with lignin and various types of whey and lactose in dry powdered form. Whey and lactose were superior to lignin and molasses in several ways: (1) the briquettes formed were much harder and denser after drying at 35–66 °F, as evidenced by their ease of handling, (2) the powdered whey/lactose materials were more easily introduced into the mixing process, and (3) whey/lactose do not require heated storage containers as lignin and molasses do. (Condensed whey lactose is also about one-half the cost of molasses.)

Pertinent Chemical and Physical Properties of Lactose. The chemical and physical properties of lactose have been comprehensively reviewed by Clamp et al. (1961) and Nickerson (1965). A discussion of some of its chemical and physical properties will be presented as they relate to this extrusion process (Table I).

In the use of lactose as an industrial binder for the extrusion process, several process conditions will exist that

Table I. Physical Tests and Chemical Analyses

sample	drop test	crushing load, lb	% Fe total	% SiO ₂	% FeO	% Fe ₃ O ₄
(1) "as received" air-dried pellets	19 (at 12 in.)	42.4	67.2	2.97	21.6	69.9
(2) prelim. fired pellets, 1 lb, 1288 °C		1360	66.6	2.5	0.5	
(3) prelim. fired pellets, 1 lb, 1260 °C		1222	66.1	2.9	Tr	
(4) final fired pellets, 30 lb, 1260 °C	20+ (at 6 ft) ^a	1092 av.				

^a No breakage, no fines.

could influence the chemical properties of this carbohydrate. These conditions would be the alkali, acid, and temperature environments. While the authors do not have any specific experimental data relative to the extrusion process and its affect on the chemical properties of lactose, Clamp et al. (1961) do report the findings of other investigators who have subjected lactose to environmental conditions similar to those encountered in this process.

One of the frequently encountered conditions in the extrusion process is the alkaline environment. Under alkaline conditions, it has been shown that lactose will undergo an hydrolysis step resulting in the release of D-galactose and other isomerized products. Specifically, in the presence of ammonium hydroxide and at a temperature of 37 °C, this hydrolysis and isomerization can occur (Hough et al., 1953).

Another process condition encountered is that of the acid environment. Depending on the strength of the acid used, lactose may either be hydrolyzed or degraded to humic substances (Takahashi, 1944, 1948). Under the conditions of this process, most likely some decomposition of the lactose could occur, resulting in the formation of a humic mass.

If lactose is subjected to a temperature of 185 °C and is held under a 4–7-mm atmospheric pressure for 10–12 h, an anhydrolactose [1,2-anhydro-4-O-(β-D-galactopyranosyl)-D-glucose] will form with the loss of one molecule of water per molecule of lactose (Pictet and Egan, 1924). Thus, heating conditions of this process could influence the formation of other carbohydrate derivatives.

The lactose physical properties of importance to the extrusion process are solubility, melting point, and heat of combustion. Most process formulations will require between 5 and 10 lb of lactose per 100 lb of finished mix. At an admixture moisture level of between 14 and 17%, a lactose working solution of 25% could be used to blend the other dry raw materials. The solubility of lactose at 25 °C is 21.6 g/100 mL of water. As the extruded products are subjected to curing temperatures of between 205 and 315 °C, the melting points of lactose become important. According to Nickerson (1965) the α form of lactose melts at 202 °C and the β form decomposes at 252 °C. Hence, as the drying/curing step proceeds, lactose loses its identity as decomposition of the molecule occurs.

Heat of combustion becomes important as the production of fuel briquettes is considered. Recognizing the presence of both the α and β forms of lactose, a combined heat of combustion of 3880 cal/g would be contributed by the lactose fraction in the briquette (Nickerson, 1965).

Little is known about the functional aspects of lactose as a binder in the extrusion process discussed herein. However, it is suggested that lactose aids in the cohesion of the particle mass during the extrusion step. Also, it is theorized that the lactose molecule aids in the molding of structural bonding which is thought to be taking place during the blending and curing/drying steps. Further investigation is needed to document these speculative concepts.

Potential in the Iron/Steel Making Industry. (1) As a new, energy conservative, lower-cost method of

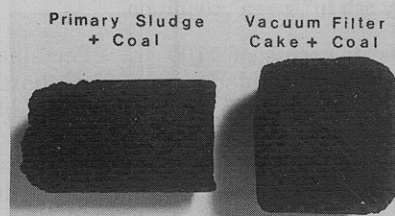


Figure 4. Sludge and coal, room temperature.

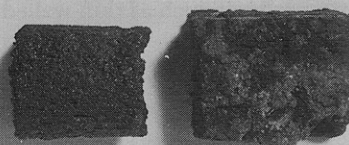


Figure 5. Rimming additive, 200 °F.

pelletizing iron ore at the mine site. The most effective means of technology transfer to the private sector appears to be through licensing to one or more large equipment manufacturers/turnkey plant constructors that design and build pelletizing plants. (2) As a means of agglomerating iron-rich "fines" (or dust) which are currently being discarded (at a cost) after collection in air pollution control devices at the steel manufacturing complexes. This aspect of the process could yield large economic returns if recycling plants were constructed at the mill, long-term operations contracts established, and iron/steel dusts processed on site and sold back to the steel mill as feed stock for the furnaces.

Recent Developments and New Areas of Experimentation. (1) Sludge from the Indianapolis Municipal Waste Treatment Plant was mixed with variations of the whey/lactose binder formulation and extruded both alone and in combination with refuse coal dust. The resultant briquettes (Figure 4), after being air-dried at room temperature, were tested in a bomb calorimeter to determine their higher heating value. In comparison to 11000–12000 BTU/lb for coal these sludge briquettes ranged from 9500 BTU/lb for vacuum filter sludge to over 11 000 BTU/lb for an oil-grease sludge/coal combination. As the City of Indianapolis currently uses almost 2 million gallons of no. 2 oil/year to incinerate their municipal sludge, an economic analysis is to be undertaken to determine the cost effectiveness of extruded sludge briquettes as an alternate fuel.

(2) As steel ingots are cast, varying materials known as rimming additives are introduced into the molten steel to achieve the desired properties. The industry has long sought a method of briquetting these materials for shipment and convenient process handling. A 2-ton sample of a calcium-sodium fluoride combination (Figure 5) was recently processed and is undergoing test at a steel mill.

(3) Light weight aggregate for use in concrete cement products can be produced from fly ash collected at coal burning utility plants. The fly ash is mixed with the binder formulation, extruded and then rapidly subjected to

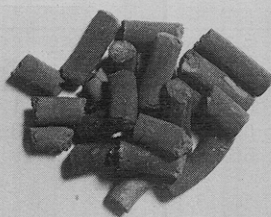


Figure 6. Fly ash in "green" condition.

temperatures in excess of 1100 °C. The small pellets (Figure 6) literally explode into coral hard, porous structures, similar in shape to popcorn. Use of this material as a concrete aggregate should reduce the steel required in high rise structures, improve the traction of road surfaces, and permit the use of light weight concrete blocks.

(4) Use of this binder, in combination with hydraulic portland cement, permits the extrusion of a wide variety of high strength products such as thin-walled pipe and tile, brick slabs, and blocks.

SUMMARY

From the diversity of the product application and the number and size of the affected industries, an insight can be gained into magnitude of the development programs that must be continued or undertaken if, in fact, a large-scale use of whey and lactose is to be developed. Further, not only must details of the processes and

products be delineated, but the economics of each must be closely examined to establish market feasibility.

For these reasons work was concentrated in the iron/steel applications as the savings in energy and resource recovery were perceived to be appreciable. Continued and expanded support is being sought from interested companies and industry associations to further and accelerate these efforts.

LITERATURE CITED

- Clamp, J. R., Hough, L., Hickson, J. L., Whistler, R. L., *Adv. Carbohydr. Chem.* **16**, 159 (1961).
 Hough, L., Jones, J. K. N., Richards, E. L., *J. Chem. Soc.*, 2005 (1953).
 Humphrey, C. W., U.S. Patent No. 3 567 811, March 2, 1971.
 Humphrey, C. W., U.S. Patent No. 3 765 920, October 16, 1973.
 Humphrey, C. W., U.S. Patent No. 3 857 715, December 31, 1974.
 Nickerson, T. A., in "Fundamentals of Dairy Chemistry", Webb and Johnson, Ed., Avi, Westport, CT, 1965, p 224.
 Pehlke, R. D., *Iron Age*, **73** (June 21, 1976).
 Pictet, A., Egan, M. M. *Helv. Chim. Acta* **7**, 295 (1924).
 Takahasi, T., *Nippon Nogei Kagaku Kaishi* **20**, 553 (1944); *Chem. Abstr.*
 Takahasi, T., *Nippon Nogei Kagaku Kaishi* **42**, 8166 (1948); *Chem. Abstr.*

Received for review May 23, 1978. Accepted April 30, 1979. Presented at the 174th National Meeting of the American Chemical Society, Division of Agricultural and Food Chemistry, Chicago, IL, Aug 1977.

Nutritional Aspects of Refeeding Cattle Manure to Ruminants

William L. Johnson

Cattle excrete manure dry matter at a daily rate of about 1% of body weight. In intensive confinement management systems proper manure disposal may be a major cost item; recovery of residual utility is therefore desirable if it can be done economically. Evidence that cattle manure does have residual nutritional value and that refeeding can be safe and economical is reviewed in this paper. Factors which influence the energy and protein value of manure or its derived products are discussed, including the influence of the ration originally fed to animals from which manure is collected and the type of processing which the manure may undergo. Other considerations for the formulation of rations with manure or manure products are palatability, the nutrient requirements of the recipient animals, disease and parasite transmission, presence of undesirable residues, performance of recipient animals, quality and consumer acceptance of the meat or milk produced, the cost of recycled nutrients compared to conventional feed sources, and the legal status of manure-derived feedstuffs.

The biological phenomenon of coprophagy is a normal act of many mammalian species. Scavenging is an even more broadly observed biological feature; in fact, until very recently in the United States, and even today in many other regions of the world, domesticated food-producing animals have received part of their nourishment by scavenging from partially digested fecal residues of other species.

Within the past decade there has been considerable research activity on the refeeding of animal and poultry excreta. Much of the impetus for such research has come from environmental concerns, which have forced the

modification of certain pollution-causing traditional waste disposal methods. The influx of nonagricultural population into rural areas, the concentration of more animal units into feedlots or confined housing, the reduction of a surrounding land base for waste disposal, and higher labor and energy costs for handling animal wastes are other reasons why there is now a greater economic incentive for the maximum utilization of animal wastes.

Adult cattle excrete one-third to one-half of the dry matter (DM) which they consume. On a daily basis the average cow excretes a quantity of DM equivalent to about 1% of her body weight. For dairy cattle, this means that about 1 kg of fecal DM is excreted for every 3 kg of milk produced, which amounts to about 1125 metric ton of wet manure per 100 cows per year. In a feedlot for finishing beef cattle, about 2 kg of fecal DM will be produced for