

# Phosphate-Bonded Ceramic–Wood Composites: R&D Project Overview and Invitation to Participate

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## **Abstract**

We are developing chemically bonded ceramic phosphate binders for the production of biofiber-based composite materials. These binders promise to have better processing and properties than some current cement and polymer resin binder systems. The ceramic phosphate binders (termed Ceramicrocrete), if used in place of cement and polymers, will significantly reduce the overall consumption of energy, curtail detrimental environmental effects compared to existing processes, and produce wood-based composite products with enhanced fire resistance, mildew resistance, dimensional stability, and durability. We are investigating the use of these binders to develop value-added products from forest thinning wood waste, treated wood waste, wood processing residue, and primary pulp and paper mill residue.

## **Background**

The project described in this paper deals with developing biofiber-based composites with novel phosphate binders for energy-efficient replacement of some current binder systems, such as cement and polymer resins. The concept of phosphate binders grew out of comprehensive work conducted at the Argonne National Laboratory with funding from the U.S. Department of Energy (DOE). The phosphate binders were developed for stabilization of radioactive and hazardous waste streams in the DOE complex, but they are now finding use in architectural and construction products, as oil field cements and bioceramics. The substitution of these binders for cement and polymers will significantly reduce the overall consumption of energy and greatly curtail the environmental effects of processing, production, and disposal. New phosphate-bonded fiber materials to be developed in the project will offer extended service-life compared with that of current building products, reduce maintenance costs to users/owners, and have greater durability.

To develop suitable binders and test them at the laboratory to actual production scale, a core team of R&D partners was formed. The partners are the Forest Products Laboratory of the USDA Forest Service, Argonne National Laboratory, the Natural Resources Research Institute of the University of Minnesota at Duluth, and the Energy Center of Wisconsin. Argonne National Laboratory invented the binder. The Natural Resources Research Institute has been working on product development with paper mills. The Energy Center of Wisconsin, a nonprofit organization, is involved in transferring the technologies from laboratories to industries. Also included in this effort is a major industrial partner, who is in the forefront of the production of fiberboard and owns three production plants.

After the binders are developed at the bench and pilot scale, full process testing will be conducted in commercial plants. In addition, several paper mills have agreed to participate in various ways, including consultation, advice, and provision of raw material for testing. Thus, the R&D team has the capability to conduct fundamental research on the binder system, produce products, test products at the industrial scale, evaluate the market for products, and conduct energy and environmental audits for products.

## Research Concept

This development project aims at developing 21st century value-added composite wood products. We propose to combine the unique properties of chemically bonded phosphate cements with those of wood fibers—virgin and recycled—to develop building products resistant to moisture, decay, fire, and mildew.

To address these needs, we have assembled a collaborative multi-disciplinary research program. The goals of this program include reduction in energy costs and promotion of resource sustainability by finding new value-added uses for low-value agricultural, woody, and recycled bio-fiber, recycled treated wood, and wastepaper mill residues.

This collaborative work using novel phosphate binders will achieve the following:

- Substantial reduction in process energy and environmental impact in production of wood composite products utilizing novel binders
- Fire- and mildew-resistant wood composite products
- Retrofit processes to adapt to current industry and practices

## Phosphate Binders

Preliminary investigations have shown that phosphate binders are potential inorganic adhesives for developing wood composite products. They may also be used to produce value-added products by recycling high volume industrial waste, such as wastepaper and pulp mill residue. Most of the work at the Argonne National Laboratory is based on the use of two types of binders. The first, Ceramicrete, has a resultant structure of  $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$  [Jeong and Wagh, 2003], which is formed by reacting magnesium oxide and monopotassium phosphate. The second binder is based on newberyite ( $\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$ ), which is formed by the reaction of MgO with phosphoric acid solution [Wagh and Jeong, 2003]. Using these two binders, Argonne has shown that several industrial high volume waste streams can be incorporated in a phosphate system. This has led to some industrial products, such as road patching material for use in winter and encapsulation material for radioactive waste and nuclear shields. Details may be found on the Argonne web site at <http://www.techtransfer.anl.gov/techtour/ceramicrete.html>.

The phosphate binders are formed by acid–base aqueous reaction between a divalent or trivalent oxide and an acid phosphate or phosphoric acid. The reaction slurry hardens rapidly, but the rate of setting can be controlled. With suitable selection of oxides and the acid-phosphates, a range of binders may be produced. The acid–base slurry is very dilute and wets most materials, except plastics, and can be used as inorganic adhesive to produce wood fiber composites. Once the slurry hardens, it binds the fibers together and produces a composite that has mechanical properties superior to that of conventional polymer or cement-bonded composites. The binder reactions can be accomplished at near room temperatures. The slurry has a near neutral pH and hence a range of industrial waste streams, such as paper mill residue, can be incorporated in the production of value-added products.

Production of phosphate binders consumes much less energy when compared to conventional binders such as cement and polymeric resins (e.g., phenol formaldehyde and isocyanate (MDI) resins) [Athena, 1993]. Since these binders are made of common phosphate fertilizers, debris from disposed products will not be detrimental to soils. In some cases, this debris may enrich soil.

Phosphate binders set rapidly at ambient temperature. Heat curing is not necessary for some phosphate adhesives that set at room temperature, unlike polymers or conventional cements, and in most cases a humid environment is not needed. The setting rates can be controlled with suitable selection of oxides and acid-phosphates. Thus, phosphate binders can be adapted to processing conditions currently used by the wood products industry.

Phosphates may be used in three ways to manufacture wood-based composites:

- a) Phosphate adhesives—The reaction slurry resulting from the acid–base reaction may be used as an adhesive similar to the current polymer resins. Thus, phosphate adhesives can be used to coat individual fibers and form a composite by binding the fibers to each other. These adhesives will behave much like current polymer resins and may be used with existing equipment. The binder content in a product is expected to be low, typically 15%–20 % by weight; therefore, phosphate adhesives have very good potential to replace current polymer-based products.
- b) Phosphate cements—Phosphate binders can be used to produce bulk composites. When conventional cement is used in fiber-based products, typical cement loading is approximately 30% or higher; phosphate cements may be used in a similar manner. The slurry formed by the acid–base reaction may be mixed with fiber or any other extender to produce solid composites [Jeong and Wagh, 2003].
- c) Surface augmentation materials—Phosphate binders may be used for coating wood panels to enhance surface properties. The phosphate slurry is very smooth; thin (<1 mm) coatings can be applied, suitable for providing fire- or water-resistance.

This project will take advantage of all three features of phosphate binders in developing products of commercial potential from waste or under-used wood resources.

### **Process Advantages With Phosphates**

The existing fabrication processes for cementitious products are energy intensive. The fiber–cement materials are currently manufactured using many different methods; the Hatschek [FCM, 1998] process is one of the most common and is considered the state-of-the-art process. A typical fiber–cement panel made under this process starts with wood chips that are broken down and mixed with water and cement; other additives are added to form a pulp-like, cellulose-rich slurry. This slurry is fed into a sieve cylinder sheeting machine. The rotating cylinder skims off a small volume of the slurry and deposits it in a thin layer onto a production surface that is placed on the forming roller. More layers are added until the desired thickness is achieved, at which point the sheets are cut to size and sent to a pre-cure chamber where they are pressed to remove water. The sheets then enter

**Table 1. Comparison of phosphate, cement, and polymer binders**

| Parameter                     | Binder            |                   |           |
|-------------------------------|-------------------|-------------------|-----------|
|                               | Phosphate         | Cement            | Polymer   |
| Minimum binder loading (wt %) | ~15%              | ~30%              | ~3%       |
| Process time                  | Short             | Long              | Short     |
| Product weight                | Medium            | Heavy             | Light     |
| Heat curing                   | May be avoided    | Not needed        | Needed    |
| Throughput                    | High              | Low               | High      |
| Mechanical properties         | Superior          | Moderate          | Moderate  |
| Water absorption              | Low               | Low               | High      |
| Fire resistance               | Good to excellent | Product breakdown | Very poor |
| Mildew resistance             | Excellent         | Good to excellent | Poor      |
| Disposal concerns             | None or minimal   | Moderate          | High      |
| Energy consumption in binder  | Low               | High              | Very high |

a pre-cure bath of 60°C (140°F) and 95% humidity environment for 8 h where the cement is further hydrated. At this point, the sheets are pressed, if necessary, and air-cured or autoclaved. Air curing involves stacking the sheets and storing at ambient conditions for 3 to 4 weeks. Autoclaving cures the sheets with high-pressure steam. Typically the products are cured for 12 h in an autoclave at 177°C (350°F) and 1.0 to 1.2 MPa (150 to 175 psi).

Many of these processing steps may be avoided through the use of phosphate adhesives, which are rapid-setting binders. Mechanical refinement of the wood chips such as flaking, screening, and water fibrillation may be avoided entirely, because the wood chips do not need to be dried. Paper mill residue is already a cellulosic slurry and is suitable to be mixed with the phosphate binder as is. Energy-intensive processes such as pre-curing and autoclaving are avoided, and, if need be, the residue-based materials may be simply air cured. Otherwise, the phosphate forming process is very similar to the current processes for cementitious products, and hence existing wood–cement or wood–composite facilities should be able to produce phosphate-bonded composites.

### Expected Product Performance

The proposed phosphate-bonded products have the potential to significantly outperform current products in the market. Previous work at Argonne National Laboratory has shown that phosphate-bonded products are superior in mechanical properties to conventional cement or polymer-based products. The density of the phosphate binder is ~30% less than that of cement. Thus, phosphate-bonded products should be stronger, lighter, nonflammable, not affected by humidity, and not vulnerable to insect attack compared to current products. Phosphate, cement, and polymer binders are compared in Table 1.

In recent processing research at the Forest Products Laboratory, particleboard furnish (pine planer shavings and sawdust) was used as a raw material for a set of baseline experiments. Using one-third phosphate binder and pressing to three densities, an extremely wide range of material properties is possible (Table 2); these properties compare favorably with those of existing products [Wolfe and Gjinolli, 1996]. Processing

**Table 2. Simple mechanical properties of phosphate-bonded wood composite**

| Parameter                       | Composite material density           |                                      |                                      |
|---------------------------------|--------------------------------------|--------------------------------------|--------------------------------------|
|                                 | Low                                  | Medium                               | High                                 |
| Phosphate binder                | 1/3                                  | 1/3                                  | 1/3                                  |
| Process pressure                | 0.0069 MPa (1 psi)                   | 0.0965 MPa (14 psi)                  | 2.69 MPa (390 psi)                   |
| Density                         | 0.60 g/cc                            | 0.80 g/cc                            | 1.25 g/cc                            |
| Flexural stiffness <sup>a</sup> | 172 MPa (0.025 ×10 <sup>6</sup> psi) | 290 MPa (0.042 ×10 <sup>6</sup> psi) | 1096 MPa (.159 ×10 <sup>6</sup> psi) |
| Bending strength <sup>a</sup>   | 0.61 MPa (88 psi)                    | 1.28 MPa (185 psi)                   | 6.53 MPa (947 psi)                   |

<sup>a</sup>Properties were obtained from specimens with short spans, so some small reduction due to shear effects may be presumed.

options for this fast-setting binder are similarly broad. Use of continuous pressing or batch pressing is plausible for the short retention time and low pressure required to create viable product properties.

The equipment required for manufacturing phosphate-bonded biocomposite products should be the same as that for current operations. Thus, existing manufacturing facilities can adapt to the phosphate processes at a comparatively low cost and without permanently committing space resources or altering existing facility geography.

Preliminary research and early prototypes indicate that phosphate-bonded products are at least as durable as current commercial wood–cement products and require little maintenance. Phosphate-bonded products have superior impact, thermal, and crack resistance and are more tolerant to weather fluctuation.

Several hurdles may be anticipated with novel binders. Inadequate wetting of fibers or wood surfaces seems to cause poor bonding. We are investigating the use of surfactants and wetting agents to achieve adequate bond formation. It is also possible that if the wood and fiber are too dry, they will absorb too much water, thereby drying out the slurry prematurely and hindering the acid–base reaction that sets it. The solution will require developing suitable control technology for reaction time and moisture content of the wood feedstock. Another hurdle may be the springback effect, which can weaken the phosphate binder matrix. This issue is being addressed by developing rapid-setting binders formulated to harden within the short duration of pressing. Finally, wood, fiber, and other residues (especially primary pulp and paper mill residue) are highly variable in properties. Thus, each batch may have different physical, mechanical, and chemical characteristics. Determining the effect of this variability on the ultimate products will ensure that the products, regardless of feedstock, have consistent performance.

## Wood Fiber Sources

Wood fiber for phosphate-bonded composites will be obtained from a wide range of forest product waste streams. Both continuing and future research is directed toward technologies for recycling wood, extending the life of wood products, and understanding energy efficiency in the production and use of forest and paper products relative to competing products. Wood-based composites have become one of the most popular and

versatile building materials. In 2001, U.S. residential structures constituted 9 billion m<sup>2</sup> (~98 billion ft<sup>2</sup>) of living space and used ~39% engineered wood composites and 40% solid wood products [Howard, 2001]. Many of these building materials are non-sustainable or require too much non-renewable energy to produce and are thus detrimental to the environment, or they are not sufficiently durable when challenged by moisture or fire. Improving the long-term durability and fire resistance of composites is critical.

The development of new technologies for the use of wood waste and paper mill residue promises to

- substantially reduce energy requirements in manufacturing of products and disposal of residue,
- penetrate new markets by creating novel phosphate-bonded products from wood waste and paper mill residue and create a diversified revenue stream for wood industry and paper mills,
- improve American competitiveness in the wood, pulp, and paper industries by eliminating the financial burden of waste disposal, and
- reduce the environmental impact of current wood composite materials and papermaking process.

The proposed binders are formed from an acid–base slurry that wets wood or other biofiber and, once set, forms inorganic-bound bio-composites. Thus, their application in wood composite production should be similar to that of current thermoset and thermoplastic polymer resin adhesives, which will allow introduction of these binders in existing production lines with minimal modifications. Preliminary trials with the binder developed at the Argonne National Laboratory have shown that the products can use high loadings of recycled and residual wood fibers, and exhibit superior binder characteristics such as fire, insect, moisture, and mildew resistance. This project will develop the necessary range of such binders to incorporate various feedstock fibers and particles available for value-added wood composite products.

Wood fiber for phosphate-bonded composites will be obtained from forest thinning wood waste, treated wood waste, wood processing residue, and paper mill residue.

### **Forest Thinning Wood Waste**

Recent surveys of the 200 million hectares (500 million acres) of productive forest land in the United States indicate that the biomass of small trees and shrubs is increasing at an average rate of 6.7 m<sup>3</sup>/s (237 ft<sup>3</sup>/s) [Sebelius, 2002]. This increase in small trees and brush in turn creates a ladder-type fuel structure that can lead to high-intensity fires. For example, in 2000 and 2002, dense, overstocked forest stands contributed to fires that burned more than 2.8 million hectares (7 million acres). Composites present a potential use for small-diameter and low-valued forest thinnings. Forest thinnings are especially

attractive for phosphate-bonded composites as they can be used in their wet/undried state and would only require energy to transport, chip, or fiberize, depending on the application.

### **Treated Wood Waste**

Limited information is available on the ultimate fate of wood waste treated with chromated copper arsenate (CCA). By the year 2006, the disposal of CCA-treated wood in Florida has been predicted to reach 1.2 million tons (MT) [Florida Institute on Phosphate Research, 2000; Gabriele and Townsend, 1998]. Nationwide, the quantity of this disposed wood is estimated to be >18 MT. Chemical treatments and the cost of collection make recovery and utilization of much of this material very difficult. Concerns have been raised about the potential environmental impact of both disposal and incineration of CCA-treated wood. The encapsulation of CCA-treated material in a phosphate-bonded composite provides an alternative to these disposal practices.

### **Wood Processing Residue**

Primary wood processing plants, such as sawmills and plywood mills, generate large quantities of wood residues in many forms, such as bark, chips, sawmill slabs and edgings, sawdust, and planer shavings. In 1991, 26 MT of bark and 74.5 MT of wood residues were generated by primary timber processing facilities in the United States [Powell et al., 1993]. This waste stream is typically an undried fiber resource and is well suited for production of phosphate-bonded products, which use moisture in the fabrication process. Another source is recovered wood from building construction and demolition waste. Contamination of this wood makes it difficult to recover and recycle.

### **Paper Mill Residue**

U.S. paper mills annually produce 8.2 million wet tons of residue [NCASI, 1992]. While the residue from each mill has unique components, it is ~50% solid. In the phosphate process, the stoichiometric water needed for the cementing reaction can be the water inherent in the residue itself. This will reduce or eliminate the use of fresh water in composite production.

## **Domestic and Worldwide Technology Status**

Inorganic-bonded materials are gaining significant market share in the United States from traditional polymer-bonded wood composites. This transition is primarily due to the improved properties (moisture stability, durability, resistance to fire and insects) of inorganic-bonded materials. However, exterior inorganic-bonded product technologies are primarily cement-based, which presents market resistance because of heavier weight and difficulty with cutting compared to polymer-bonded wood composites. Cement-based panels have been in common use in Europe for the past two decades as part of building code efforts to improve fire performance and durability. Conversely, until relatively recently, U.S. codes had not enacted stringent requirements for fire performance and durability of composites, and wood-based products were used as the baseline. In the past 5 years, the adoption of International Building Codes in many jurisdictions has prompted

builders and contractors to seek materials with enhanced performance [Nisbet and Venta, 2000].

In domestic markets, where gypsum products have dominated, the cement/gypsum blend has provided a more durable product for wet-use applications and for fire partitions in multi-family structures. A gypsum–fiber composite has also been successfully introduced. Efforts to reduce weight or increase stiffness include incorporation of low-density paper fiber, perlite, or styrene foam beads throughout the panel cross-section or in the core of a three-layer construction. These gypsum enhancements provide better structural performance to resist impact and lateral forces (e.g., flooring applications or fire hose blasts during fire testing) and reduce creep deflection. Markets for improved gypsum–fiber composites are single-layer flooring and tile-backing boards [Shook, 2000].

Primarily as a consequence of developments in Europe, U.S. equipment manufacturers have fully developed all the process line equipment needed for cement board production. Technology for embossing and color finishing of these panels is also well established. The continuous production lines and short haul distances to markets have made these plants feasible and profitable in Europe. Weight is still a concern and some movement toward gypsum/cement blends is occurring in European siding markets. Enhancing material stiffness with wood fiber/particles is common; for some applications, the use of glass fibers allows some reduction in overall product thickness or weight.

It is only in the last 10 years that phosphate binders were discovered and extensive research initiated. Their full potential has not yet been realized. Most of the work has been directed toward applications in fields such as radioactive waste stabilization. As a result, much of the wood industry is not aware of phosphate binders and their potential in composite wood products. The composite wood product industry has already made significant capital/plant investment, and being very conservative in its outlook, rarely takes risk in new technologies. This project will provide an opportunity to demonstrate the enhanced product properties, energy savings, and environmental benefits of phosphate binders to the composite products industry.

## **Project Goals and Scope**

The goal of this research program is to develop wood fiber composite products bonded with inorganic phosphate binders. Fundamental relationships between materials and processes will be developed, and this understanding will be applied to product manufacture. Use of low-grade fiber and recycled materials, integration of novel binders in the existing production equipment and processes, and reduction of energy consumption in processing will promote a broader goal of sustainable development and U. S. competitiveness.

The project will focus on the following products:

- a) Durable exterior products—Siding, decking, roof systems, sheathing, and other

products that require superior moisture and decay resistance.

- b) Fire-resistant products—Fire door cores, elevator shaft and stairwell liners, and interior wall sheathing, which will be enhanced by improved resistance to flame spread.

## **Objectives and Methods**

The first year of this 3-year project will be devoted to bench-scale investigations of adhesive properties of various phosphate binders with wood fiber and optimization of properties such as density, viscosity, time and setting temperature of acid–base slurry, and bond strength with different types of wood. This information is crucial to bench-scale production of typical composite wood products and optimization of processing parameters. During the second year, medium-scale wood products will be produced in participating laboratories and tested for standards requirements. The focus of the third year will be on pilot-scale testing, energy economics of production, and quantification of environmental benefits for the industry and consumers. All work will be conducted in close collaboration with participating industrial partners, culminating in demonstration and implementation of the technology.

The goal of the first year is to develop production process parameters and resultant material characteristics. The objectives include selection of phosphate binders that are compatible with various fiber types and processing requirements, fabrication of bench-scale samples, and optimization of binder content. We will also construct predictive models of performance for targeted fire- and moisture-resistant products.

Work in the second year will be at the pilot scale, aimed at further defining material compatibility and processing requirements. Pilot-scale product trials and evaluations of medium-scale (0.6- by 0.6-m, 2- by 2-ft) panel products will be conducted in participating laboratories and tested for standards requirements. We will also evaluate superior process parameters for production-scale development. Issues to be addressed are energy consumption, production throughput, and disposal of effluents such as acid water, if any. The products identified by pilot-scale testing will be subjected to testing with ASTM standards procedures to develop a database on engineering properties.

The objective of the third year is to transfer the knowledge gained to the participating industries through production-scale trials and formal technology transfer. Quality of products, production scale energy economics, waste disposal, and consumption of valuable resource such as binder and water will be monitored. In addition, packaging of binders, transportation to user site, and storage will be addressed. At the end of this year, a full information package will be developed to facilitate transfer of the technology directly for production by participating industries. Interests of the industrial partners will be guarded according to the collaborative R&D agreements reached between the industry and the participating laboratories. The ultimate objective will be to transfer the technology in accordance with DOE and USDA guidelines.

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