#759a – Commercialization of Nanotechnology (22006)

Decontamination, Safety Applications, and Products Based on Nanomaterials

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NanoScale Corporation is a dynamic and innovative company focused on the development and commercialization of advanced products, including our proprietary nanocrystalline metal oxides and related technologies. NanoScale works in four primary business segments: the sale of packaged and bulk high performance materials, application services related to the materials, application development contracts, and providing analytical services.

NanoScale's business model includes pro-actively identifying and pursuing mutually rewarding joint efforts with private and government sectors to provide unique solutions. In conjunction with the Department of Defense (DoD), Department of Energy (DoE), National Institutes of Health (NIH), National Science Foundation (NSF), Environmental Protection Agency (EPA), other federal agencies, and commercial customers, NanoScale has invested over $26 million in research and application development. Additionally, independent of our joint efforts with these entities, NanoScale maintains a well funded and active internal R&D program that is dedicated to advancing our Intellectual Property portfolio, identifying new scientific applications for our advanced earth minerals, identifying new markets and applications, and providing new added value products and services for our potential and existing customers.

NanoScale has a strong and ever-growing portfolio of Intellectual Properties and trade secrets. The company has ownership and exclusive rights to twenty-four (24) issued U.S. patents and seventeen (17) issued International patents, with an additional six (6) U.S. and thirty-eight (38) International patents filed and pending.

Core Technology Overview

NanoScale has developed a line of nanocrystalline materials (NanoActive® materials) with remarkably high chemical reactivity. These materials have been shown to be effective against a broad range of hazards, including Toxic Industrial Chemicals (TICs), Chemical Warfare Agents (CWAs)[1], as well as Biological Warfare Agents (BWAs). Reactive nanomaterials are non-flammable and stable at high and low temperatures.

[1] Wagner, G.W.; Bartram, P.W.; Koper, O.; Klabunde, K.J. "Reactions of VX, GD, and HD with Nanoscale MgO," *J. Phys. Chem. B.* **1999**, *103,* 3225; Wagner, G.W.; Koper, O.B.; Lucas, E.; Decker, S.; Klabunde, K.J. "Reactions of VX, GD, and HD with Nanosize CaO: Autocatalytic Dehydrohalogenation of HD," *J. Phys. Chem. B*. **2000**, *104*, 5118; Wagner, G.W.; Procell, L.R.; O'Connor, R.J.; Munavalli, S.; Carnes, C.L.; Kapoor, P.N.; Klabunde, K.J. "Reactions of VX, GB, GD, and HD with Nanosize Al_2O_3. Formation of Aluminophosphonates," *J. Am Chem. Soc*. **2001**, *123*, 1636.

Their high surface area and unique morphology give NanoActive materials very different properties compared to either bulk materials or singular atoms.[2]

Macroscopically, metal oxide nanoparticles are fine powders, but closer examination shows that nanoparticles consist of very small crystals (usually 2-10 nanometers in size) that form larger clusters. One example is NanoActive MgO Plus, which typically has a surface area of 650 - 700 m^2/g and an average crystallite size of 4 nm. By way of comparison, the specific surface area of many conventional MgO products is 20-40 m^2/g.

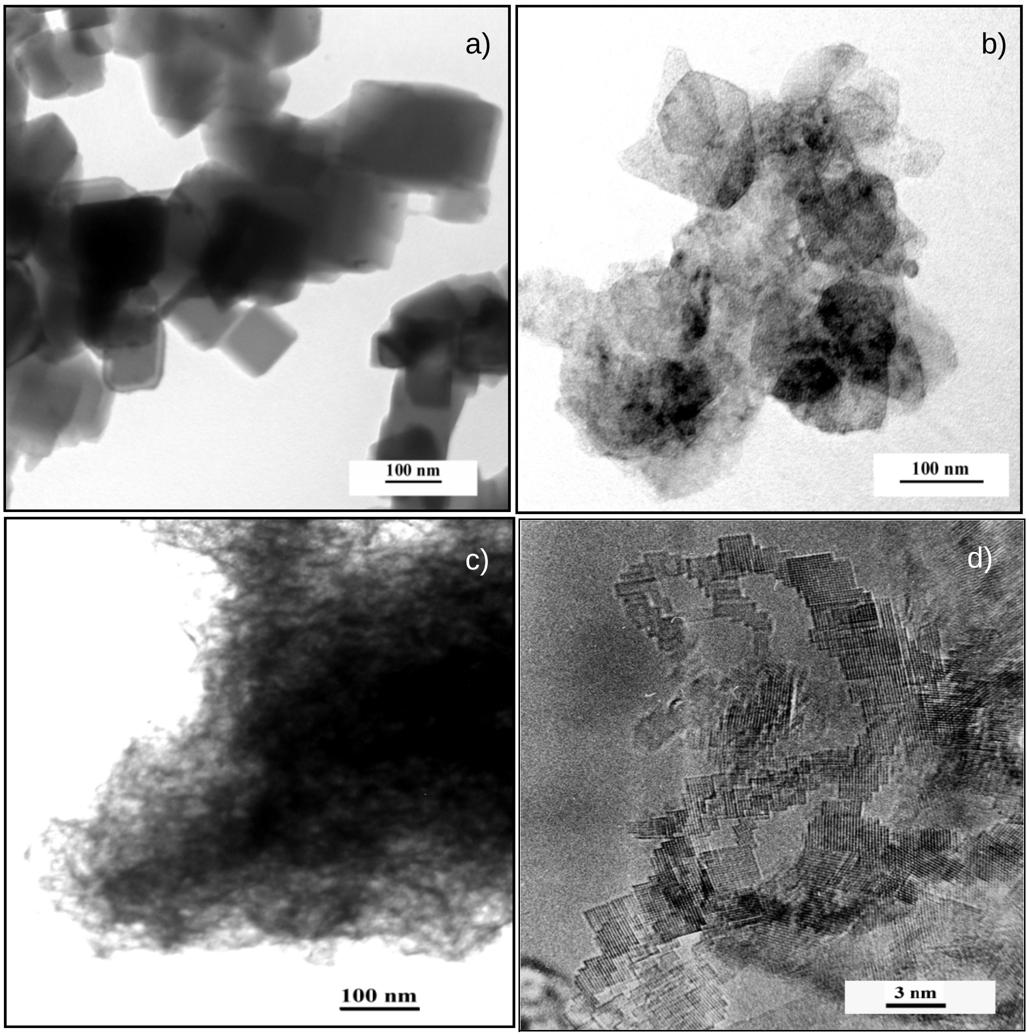

Figure 1: TEM of Magnesium Oxides a) Conventional, b) NanoActive MgO, c) NanoActive MgO Plus & d) High-resolution NanoActive MgO Plus.

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[2] Klabunde, K.J.; Stark, J. V; Koper, O.; Mohs, C.; Park, D.G.; Decker, S.; Jiang, Y; Lagadic, I.; Zhang, D. "Nanocrystals as Stoichiometric Reagents with Unique Surface Chemistry," *J. Phys. Chem. B* **1996**, *100*, 12142-12153 (invited feature article).

Differences between NanoActive products and their conventional analogs are shown by the appearance of various samples of MgO as shown in Figure 1. The Conventional sample (1a) has an appearance of cubes 60-80 nm in length while NanoActive MgO (1b) is composed of hexagonal platelets ~ 80 nm in length and 10 nm thick. NanoActive MgO Plus, (1c) and (1d), is made up of a porous web-like material. The high resolution TEM, Figure 1d, shows very small 2-4 nm cubes that aggregate into interconnected polyhedral structures, forming a highly porous material with numerous, low-coordinate corners and edge sites.

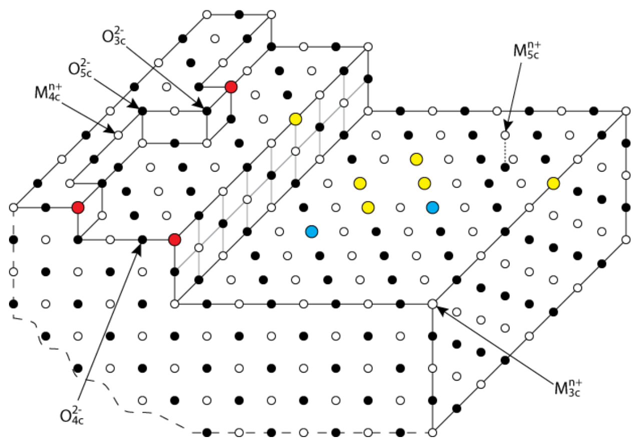


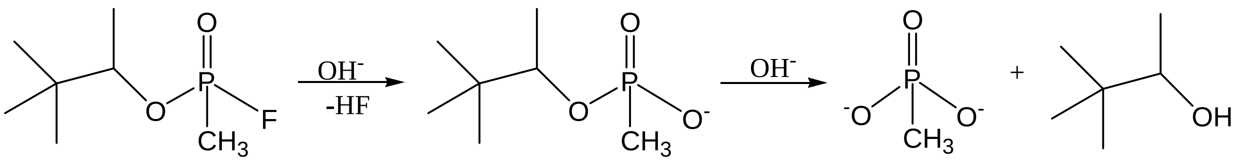
Figure 2: Schematic of Metal Oxide Surface Illustrating Surface, Edge and Corner Ions.

A conceptual view of a typicalmetal oxide nanocrystal is given in Figure 2. Cation (M^{n+}) sites in the interior of the crystal (not visible) have a coordination number of 6, surface cations (representative ions shown in blue) have a coordination number of 5, edge cations, shown in yellow, have a coordination number of 4, and corner sites, shown in red, have a coordination number of 3. A surface vacancy defect reduces the coordination number of surrounding ions; this is shown by a hole surrounded by yellow ions. The lower the coordination number of a cation site, the higher its reactivity, in general.

This unusual morphology results in enhanced chemical reactivity and suggests a two-step decomposition mechanism; the first step is adsorption of toxic agent by physisorption, followed by a second step of chemical decomposition. This two-step mechanism substantially enhances the detoxification abilities of nanocrystalline materials because it makes the decomposition less dependent on temperature. Although chemical reactivity is slowed at lower temperatures, physisorption, which is required before chemical reaction, is *enhanced* at low temperatures.

The unique characteristics of materials developed at NanoScale lend themselves to applications in the fields of chemical neutralization, odor control and water decontamination. Many materials currently used for these applications, including activated carbon, can adsorb the harmful contaminant but do not always destroy it. NanoScale's nanocrystalline materials have been proven to not only remove harmful contaminants but also chemically destroy them.

For example, testing conducted at the Aberdeen Proving Grounds elucidated the mechanism of agent destruction for chemical warfare agents. Wagner et al.[3] reported that NanoScale metal oxide nanomaterials were very effective in room temperature destructive adsorption of chemical agents, such as VX, GD and HD. The destruction products of GD (soman) are pinacolyl methylphosphonic acid (GD-acid) that converts to surface bound methylphosphonic acid (MPA), and (hydrofluoric acid) HF, which is also neutralized by metal oxides. Figure 3 illustrates this reaction.



GD **GD-acid** **MPA**

Figure 3: GD Degradation by Reactive Nanomaterials

Product Development and Commercialization

NanoScale's core technology has recognized value in the areas of containment and neutralization of CWAs and TICs, odor control, antimicrobial applications, water decontamination, as well as others. In addition to extensive research and development efforts, NanoScale focuses on the transfer of laboratory developments into commercial products for the improvement of environmental safety and overall quality of life. Current commercial offerings include FAST-ACT®, NanoActive chemicals, and OdorKlenz®, which have been sold throughout the world. Other products are currently in the development phase with both government and commercial partners.

FAST-ACT

FAST-ACT (First Applied Sorbent Treatment Against Chemical Threats), a revolutionary chemical hazard containment and neutralization system, is NanoScale's first commercial product and was developed with the end user in mind. The FAST-ACT family of products utilizes a non-toxic, non-corrosive, non-flammable dry powder that is capable of neutralizing a wide range of toxic chemicals with the added capability to destroy CWAs. FAST-ACT is sold to first responders and HAZMAT teams around the world and used in response to liquid chemical spills and vapor releases. It has significant advantage over other chemical release mitigation measures in that it can be used on any release without the need for prior identification of the contaminant. Figure 4 shows the efficacy of FAST-ACT against vapor hazards.

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[3] Wagner, G.W.; Bartram, P.W.; Koper, O.; Klabunde, K.J. "Reactions of VX, GD, and HD with Nanoscale MgO," *J. Phys. Chem. B.* **1999**, *103,* 3225; Wagner, G.W.; Koper, O.B.; Lucas, E.; Decker, S.; Klabunde, K.J. "Reactions of VX, GD, and HD with Nanosize CaO: Autocatalytic Dehydrohalogenation of HD," *J. Phys. Chem. B*. **2000**, *104*, 5118; Wagner, G.W.; Procell, L.R.; O'Connor, R.J.; Munavalli, S.; Carnes, C.L.; Kapoor, P.N.; Klabunde, K.J. "Reactions of VX, GB, GD, and HD with Nanosize Al_2O_3. Formation of Aluminophosphonates," *J. Am Chem. Soc*. **2001**, *123*, 1636.

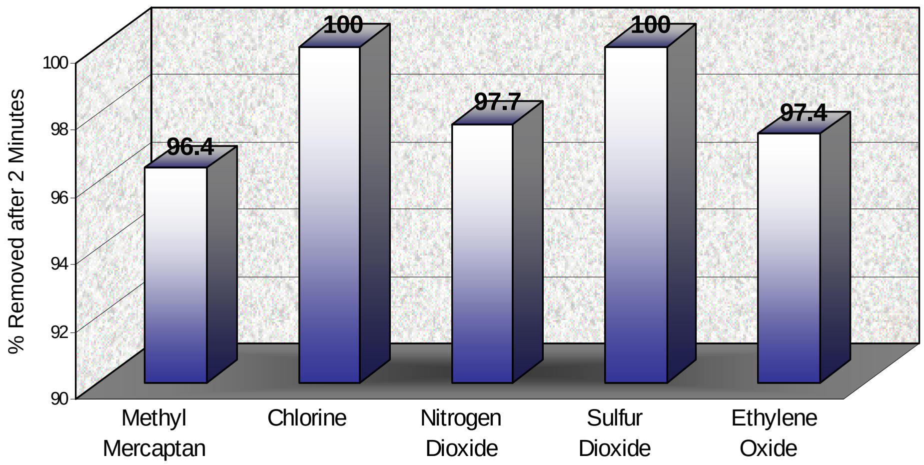

Figure 4: FAST-ACT Efficacy Against Vapor Hazards

The FAST-ACT material formulation was optimized for decontamination of a variety of chemical hazards (Table 1) and the product line includes pressurized cylinders, bulk pails and shakers. The variety of delivery methods allows a user to safely contain any size spill or leak.

Table 1: Efficacy of FAST-ACT Against Hazardous Materials

Neutralization		Adsorption	Containment	Not Protected
Corrosive Materials		Vapor Hazards	Liquid Solvent Spills	
Acids Inorganic and Organic Hydrochloric Acid Hydrofluoric Acid* Hydrobromic Acid Nitric Acid* Phosphoric Acid Sulfuric Acid* Acetic Acid Methanesulfonic Acid Ethanesulfonic Acid Benzenesulfonic Acid Toluenesulfonic Acid Phosphorus Pesticides Dimethylmethyl Phosphonate Paraoxon Parathion* Sulfur 2-Chloroethyl Ethyl Sulfide Methyl Mercaptan Phenols Nitrophenols Chlorophenols	Carbonyl Compounds Aldehydes* Ketones Carboxylic Acids Nitrogen Compounds Acetonitrile* Sodium Cyanide (aq) 4-vinylpyridine Halogens/Halides Acetyl Chloride Chloroacetyl Chloride Chlorine Chloroform Hydrogen Bromide* Cyanogen Chloride Methylene Chloride Carbon Tetrachloride TCE, PCE Bis-(2-Chloroethyl) Sulfide Pinacolyl methylphosphonofluoridate O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate	Acidic and Caustic Gases Hydrogen Chloride Hydrogen Fluoride Hydrogen Bromide* NOx/N2O4* Sulfur Dioxide Hydrogen Sulfide* Diborane* Hydrogen Selenide* Phosphine* Ammonia Carbonyl Sufide Hydrogen Cyanide* Chlorinated Organics Acetyl Chloride Chloroacetyl Chloride Chloroform Methylene Chloride Halogens Chlorine* Bromine Iodine Volatile Organics Methyl Mercaptan* Ethylene Oxide* Formaldehyde* Phosgene* Arsine*	Alcohols/Phenols Ethanol Methanol Allyl Alcohol* Nitrophenols Chlorophenols Caustics Anhydrous Ammonia* Metal Hydroxides (aq) Petrochemicals Diesel Gasoline Oils Others Acrylonitrile* Benzene Hydrazine* Toluene Acrolein* Methylhydrazine* Methylisocynate*	Biologicals Bacteria Viruses Spores Nuclear Radiological Heavy Metals Solid Waste
LIQUID & VAPOR CHEMICAL SPILLS AND RELEASES				

NanoActive Chemicals

The NanoActive line of specialty chemicals are highly porous metal oxides with unmatched surface areas and unique nanocrystalline morphologies. The distinctive properties provide exceptional chemical reactivity for a broad range of industrial, commercial, military, and research applications. The markets include decontamination, personal protection, energy, electronics, catalysts, and other advanced materials. Despite the absence of any regulatory requirements for these materials, NanoScale has utilized independent certified laboratories to conduct evaluations of its products for health and safety risks. NanoActive materials have been proven safe through oral, pulmonary, ocular, and dermal toxicology testing. Additionally, NanoActive materials have been shown to be no more toxic than their non-nanocrystalline analogues. The materials are available to markets in a variety of forms including powders, granules, suspensions, and can be customized for market specific applications.

Table 2: Toxicity Data for NanoActive Materials

	*NanoActive TiO_2**	*NanoActive MgO***
Acute Oral Toxicity	LD_{50} > 2 g/kg	LD_{50} > 5 g/kg
Acute Dermal Toxicity	LD_{50} > 5 g/kg	-
Acute Dermal Irritation	(PII) of “0” Non-irritating	EPA Category IV Non-irritating
Skin Sensitization	Non-sensitizer	Non-sensitizer
Acute Eye Irritation	Practically Non-irritating	EPA Category III Slightly irritating
Acute Inhalation	Nontoxic 2334 mg/m^3 for 4 hrs	Nontoxic 259 mg/m^3 for 4 hrs

******* *- Testing conducted at MPI* ******** *- Testing conducted at CHPPM*
PII *– Primary Irritation Index*
In all Oral and Dermal Toxicity studies no test animals died during the exposure time, therefore LD_{50} is > test limit dose for the specific NanoActive material studied.

OdorKlenz

OdorKlenz, NanoScale’s family of odor elimination products, is based on the same destructive adsorption technology. OdorKlenz has been proven commercially effective at mitigation of a wide variety of odors and is being used in areas such as the commercial and residential real estate industry, death care and medical industries, veterinary necropsy labs, and gross anatomy labs. The largest market impacted by the

OdorKlenz technology is disaster recovery. NanoScale was able to adapt the technology and configure it into a cartridge that can be used with existing industry air scrubbers for the elimination of airborne odors. The technology allows for safer and faster odor elimination.

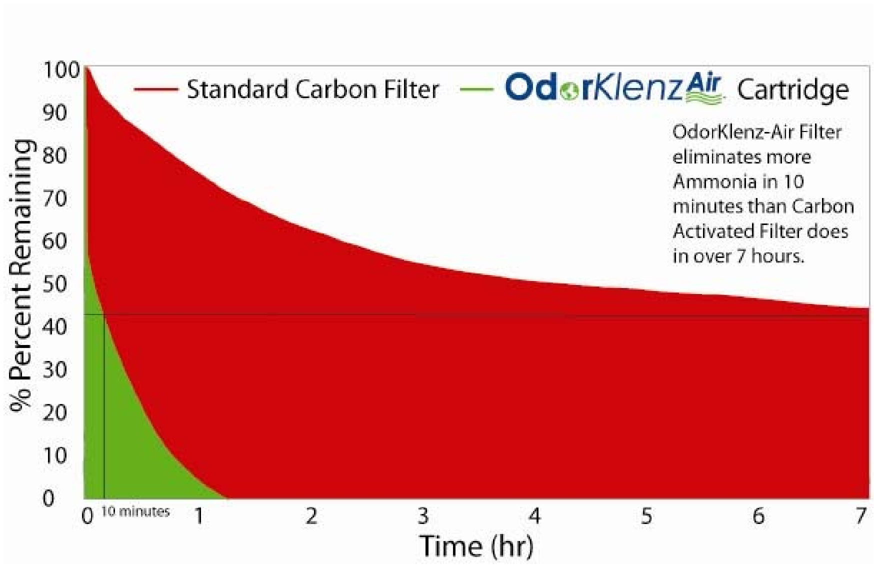

Figure 5: Ammonia Removal from Air

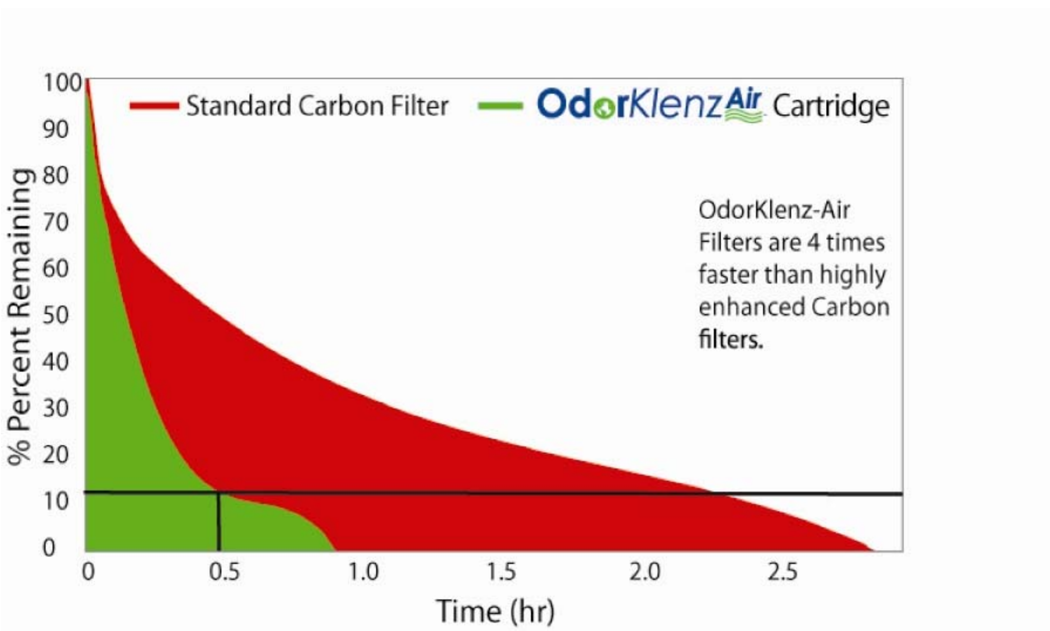

Figure 6: Hydrogen Sulfide Removal from Air