

Kaolin: From Ancient Porcelains to Nanocomposites

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Kaolinite crystals from Twiggs County, Georgia, USA. The largest crystal is 50 μm .

1811-5209/14/0010-177\$2.50 DOI: 10.2113/gselements.10.3.177

Kaolin has played a prominent role in both Earth and human cultural history, and will continue to do so into the foreseeable future. Its abundance in the rock record has varied according to the waxing and waning of life faunal radiations, crustal differentiation, and climatic conditions. Kaolin-group minerals play an important environmental role in soils of tropical and temperate regions. They also occur in hydrothermal zones, where they form at the expense of felsic rocks through acid hydrolysis, and commonly in episodic geologic events. Kaolin deposits form the basis of many modern materials, but the first sophisticated human use was in porcelains from Jingdezhen, China. From the initial discovery of kaolin and its sculptural use 2000 years ago to today's modern applications in nanocomposites, kaolin has been important throughout cultural history. Art and science are intertwined by the properties and uses of this amazingly small clay mineral.

KEYWORDS: kaolinite, clay minerals, porcelain, Jingdezhen, China, weathering, hydrothermal, hydrolysis

INTRODUCTION

Kaolin is a naturally occurring clay-rich material that has played an important role in shaping Earth history and human culture. Geologically, kaolin is simply the name for a rock dominated by kaolin-group minerals. Minerals in the kaolin group comprise some of nature's most abundant nanomaterials; their unique shapes and layered structures make them important resources in the Earth's near-surface critical zone. Murray and Keller (1993) noted that there are kaolins, kaolins, and kaolins, meaning that kaolin-group minerals are prevalent in many geologic settings. These settings include places where aluminosilicate-rich sedimentary, igneous, and/or metamorphic rocks react with water, acids, and redox-changing solutions to create halloysite, kaolinite, nacrite, or dickite, the minerals constituting the kaolin group (see Table on page 206). Kaolin-group minerals are formed in terrestrial hot springs, in hydrothermal systems, in tropical and temperate soils, and in sedimentary basins, commonly under the influence of microbiologically mediated geochemical reactions.

Worldwide mine production and reserves of kaolin amount to about 37 million tons (Mt), with the United States being a leading producer (5.3 Mt per year). Kaolin's value is nearly \$150 per ton. Compared to other commonly mined clays in the United States (e.g. bentonite and Fuller's earth), kaolin is the largest in tonnage (source: USGS Minerals Commodity Summary, February 2014 production).

Kaolin's economic significance is worldwide, as it occurs in nearly half the manufactured products in daily use. Over the past 100 years, product applications include paper coating, ceramics, rubber, plastics, pharmaceuticals, paints, and cosmetics. Kaolin differs from other clay minerals in that it promotes brightness, opacity, smoothness, viscosity, and printability. New applications include the creation of nano-sized materials for use in the inks of modern printers, nontoxic pesticides in organic farming, and polymer composites used in the packaging industry. The future holds promise for nanocomposites derived from kaolins to improve the rheological, thermal, and organic complexation capabilities

of products used in engineering and medicine. Imagine being able to delay the melting of a steel structure simply by coating it with a clay-polymer coating or to deliver DNA into a cancer cell by using a halloysite nanotube as a Trojan horse. This issue of *Elements* provides insights into kaolins and their unique properties.

Kaolin-group minerals share a basic 1:1 layer structure made of repeating sheets of silica tetrahedra and alumina octahedra; their composition is ideally expressed by the formula $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ (FIG. 1). The combination of the 1:1 structure (including its hydroxyl groups), the variability of site substitutions (e.g. $\text{Fe}^{3+} \rightarrow \text{Al}^{3+}$), and layer stacking disorders gives rise to an array of material properties that benefit countless products. In fact, if you are right now holding a paper copy of *Elements* magazine, your fingertips are likely touching kaolinite. In this article, we briefly review the role of kaolin in Earth's history, including the formation history of different types of kaolin deposits, the cultural uses of kaolin, ancient kaolin mining practices, and ancient and contemporary Chinese influences on the production of kaolin-based ceramics.

KAOLIN IN EARTH HISTORY

Clay minerals probably started forming on Earth immediately after planetesimal formation (Hazen et al. 2013). Given the presence of (1) water, (2) a source of acid, and (3) aluminosilicate rocks on the early Hadean Earth, kaolin-group minerals were likely present and were perhaps the first or second clay group to form, preceded only by smectite-group clays derived from mafic minerals. The abundance of kaolin in the rock record has fluctuated with major events in Earth's evolution (FIG. 2A), particularly as biogeochemical

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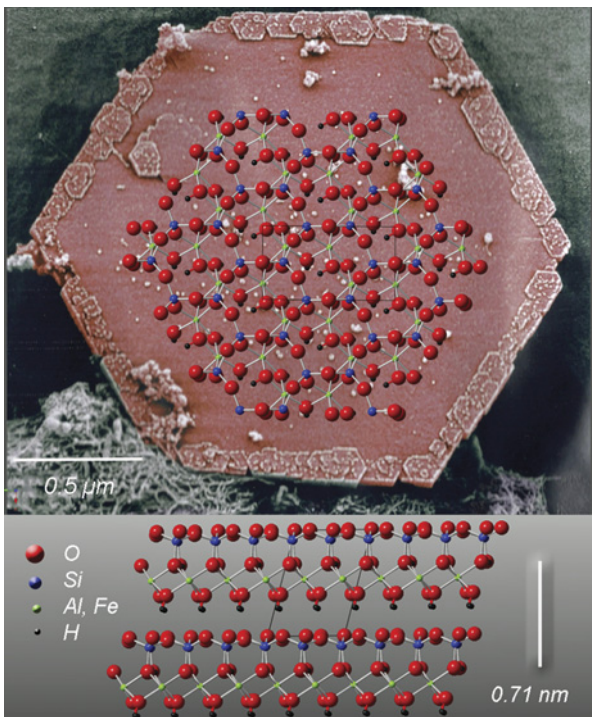


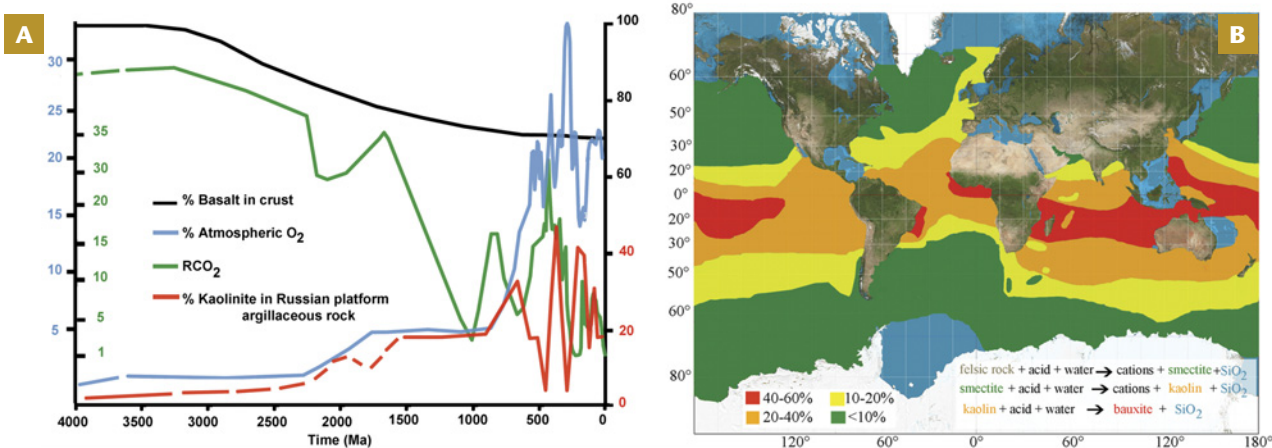
FIGURE 1 (Top) Kaolinite crystal in pseudo-hexagonal form (false-color image from a scanning electron microscope). Edges are adorned with spiral growth forms, which emphasize the multigenerational nature of kaolin-group minerals. The scale bar is 180 times smaller than the diameter of a human hair. Overlain on top of the crystal (not to scale) is the atomic structure of kaolinite, showing atom locations parallel and perpendicular (Bottom) to the sheet structure. The bottom image also shows the repeat distance of the 1:1 layer structure. The scale in lower right is about 125,000 times smaller than the diameter of a human hair.

processes on Earth have episodically transitioned their electron acceptors from CO₂ toward O₂ (Lyons et al. 2009; Shock et al. 2010). The exact timing of the first appearance of kaolin is debated; however, it is generally agreed that events such as the emergence of life during the Hadean (Hazen et al. 2008), the Great Oxidation Event during the

Proterozoic, the onset of Paleozoic faunal radiation during the Cambrian, and variations in atmospheric temperatures and CO₂ and O₂ concentrations (Berner 2006, 2009) all influenced kaolin production and preservation rates. The occurrence of kaolin-group minerals on Mars (Bishop et al. 2008) supports the notion of their early appearance on Earth. The modern distribution of kaolinite in ocean sediments shows the importance of climate. Both temperature and rainfall correlate positively with the latitudinal abundance of kaolinite relative to other clay minerals (Fig. 2B).

Kaolin-group minerals are prevalent in well-drained, temperate to tropical soils, where the reaction kinetics favor the formation of halloysite and kaolinite. The occurrence of kaolin-group minerals in soils is environmentally important (Cygan and Tazaki 2014 this issue) and although common, they do not constitute exploitable deposits. The geologic processes that concentrate kaolin-group minerals into economic deposits fall into several categories defined by the parent material and the alteration process that affected them (Murray et al. 1993). Precursor felsic rocks include granites, rhyolitic pyroclastics, and shales. The occurrence of kaolins can be viewed simply as an intermediate stage in a series of hydrolysis reactions involving aluminosilicate rocks (Fig. 2B). Hydrolysis is driven by acidic volcanic/plutonic hydrothermal fluids and/or meteoric water flow influenced by microbial metabolisms, formation hydraulic conductivity, and proximity to the surface. Most economic kaolin deposits form as a result of more than one of these alteration mechanisms. As Earth has evolved toward greater crustal differentiation (i.e. more felsic rocks) and more biological activity on its surface (i.e. more oxidation and organic acid production), the occurrence of large kaolin deposits is generally thought to have increased with time (Fig. 2A). Economic deposits of kaolin are therefore typically hosted in geologically younger rocks (Upper Mesozoic and Lower Cenozoic) and have commonly been modified by Pleistocene to Recent surface weathering (Hurst and Pickering 1997).

Economic kaolin deposits are broadly grouped into (1) primary and (2) sedimentary-rock hosted. Primary kaolins can be subdivided into those that originate in granitic rocks and those that originate in rhyolitic rocks. They are further



subdivided by the extent to which they have been hydrothermally and/or meteorically altered. China and Mexico host large hydrothermally altered granitic residues (Wilson 2004), whereas Japan hosts large hydrothermally altered pyroclastic deposits (Joussein et al. 2005). Primary deposits are located throughout the world and are enhanced by the weathering of volcanic glass, feldspar, and mica by meteoric water. These weathering processes form saprolite extending as deep as 100 m from the surface. Kaolin deposits in Cornwall (England) and Ukraine are perhaps the most widely recognized sources; their origin is attributed to the alteration of granitic rock by hydrothermal and meteoric waters (Murray et al. 1993). A feature common to all these primary deposits is a porous mixture of stack-like kaolinite and tubular halloysite that is low in iron and titania; if buried under appropriate basin conditions, such mixtures can host petroleum deposits.

Sedimentary-rock-hosted kaolins are by far the most concentrated and therefore the most important sources of kaolin ore. Production sites are located in Cretaceous and Eocene shales and sandstones in northern Brazil, the southeastern United States, southern China, and northeastern Australia. The geologic conditions needed to create these unique deposits may not have an exact modern analog; however the deposition of neritic muds proximal to aluminosilicate rocks undergoing intense chemical weathering is likely the first step toward their genesis. Conditions within the sedimentary beds are almost the antithesis of those needed for an economic oil shale deposit. Kaolin shales are extremely poor in organic matter and iron. Postdepositional

uplift of kaolin-bearing shales and sandstones results in further meteoric weathering. The small amount of organic matter and iron sulfides that may be present is oxidized by microbial action (Hurst and Pickering 1997; Shelobolina et al. 2005). During this process the remaining alkali and alkaline earths, iron, and silica are mobilized (Schroeder et al. 2004) and precursor kaolin-group minerals (halloysite and disordered kaolinite) undergo Ostwald ripening (Lowe 1991). Sedimentary kaolins are thus up to 95% pure. Anatase (a titanium-bearing mineral) and iron oxides occur ubiquitously as trace phases in sedimentary-rock-hosted deposits due to the insolubility of Ti^{4+} and Fe^{3+} (Railsback 2003). Variables that affect the final kaolin ore include the initial rock composition, the geologic exhumation history and resulting landscape position (i.e. thickness and permeability of overburden), the hydrologic flux (i.e. climate), and time. These factors conspire to make different types of postdepositional alterations, and industry selectively mines various grades of deposits, often referred to as gray, red, or cream in color and hard or soft in texture (see Kogel 2014 this issue).

KAOLIN IN CULTURAL HISTORY

The first sophisticated cultural use of kaolin dates back over 2000 years to Jingdezhen, which is situated in Jiangxi province in southeastern China. Located thirty miles to Jingdezhen's northeast is Gaoling Shan—translated literally as “high ridge”—and the first mining site for *gaoling tu*, or kaolin clay (FIG. 3). High-tech use of *gaoling tu* started with porcelain production, utilizing what is now known as china clay.



FIGURE 3 (A) Gaoling Shan is a range of mountains in the northern part of Jiangxi province of China. (B) Dongbu Village is a small community situated at one of the entrances to Gaoling National Mining Park. The quaint little village was once a hub for transporting kaolin clay from the mines to Jingdezhen. (C) Off the river small docks once awaited the white kaolin cargo (photo in local kaolin museum © National Geographic Society, 1920). (D) The Tianbao Kiln (dragon kiln), built on the

sloping hillside, is tapered at both ends. Stoking holes progressively preheat successively higher sections, allowing for careful temperature control; three days were often needed to complete a firing. (E) Sculpture of a foreigner and his horse coming from the far west to trade during the Yuan dynasty. Their expressions vividly reflect the cultural and economic exchanges taking place at that time and during the Song dynasty.

The first porcelains were a simple formula consisting of only a feldspathic china stone, or *petunze* in Chinese. *Petunze* was originally found as a weathered, top-layer powder, and the sources were abundant near Jingdezhen. The nonplastic quality of this ground stone made wheel-throwing very difficult and restricted product size. The success rate of kiln-fired pieces was low as they were prone to distortion and collapsed in the kiln due to the instability of the material when shaped into thin objects and fired at 1200°C. The history of ceramics is relatively short compared to traditional Chinese medicine, where one would never use only one herb but rather a combination of ingredients for successful treatment. Applying this principle to ceramics, using only one material when making porcelain was thought to be causing the problem. When the fired porcelain became too soft, they thought that its “yin” was too strong. Ancient Tiwu (体) philosophy focuses on results rather than theory and considers that perception is learned through observation and experience of actions. The application of this philosophy led to one hundred years of experimentation to find the balancing factor—“yang.” Near the end of the Song dynasty (1127–1279), a kiln master with the surname He conducted materials experiments in the mountains around the small village of Yaoli. Born into a poor family, Master He was adding a fine-particle-sized white material mined near the surface on Gaoling Mountain. Combining this *gaoling tu* (kaolin) with the china stone (weathered granite) gave the porcelain strength. He did not share his findings but others soon got the same results. During the Yuan dynasty (1279–1368), a dual-formula (binary) porcelain clay body made of approximately 60% china stone and 40% kaolin was developed. These percentages varied depending on the chemical makeup of the material from different mining sources. Alumina-rich kaolin is thought of as the bones of porcelain, while the silica in the china stone is the flesh. Combining the two materials brought the “yang” and “yin” together (FIG. 4).

Near the end of the Southern Song dynasty, exhaustion of the sources for top-layered china stone led to a raw-materials crisis in the porcelain industry. Mining deep into the mountains provided new sources, but artisans could not make porcelain with only this ore, due to its relatively low

sintering temperature. It was the addition of kaolin that allowed the middle- and under-layered china stone to be used, and the porcelain industry flourished. The kaolin allowed the firing temperature to rise, reducing distortion and improving the success rate and quality. The new dual-formula ware became the first true, high-fire porcelain, with firing temperatures exceeding 1300°C. This created great prosperity in the ceramic industry, starting during the Yuan dynasty (1271–1368) and continuing through the Ming (1368–1644) and Qing dynasties (1644–1911). Jingdezhen porcelain was said to be as white as jade, as bright as a mirror, and as thin as paper, and when it was struck, it made a sound as sweet as a *qing*, a musical instrument similar to a chime and made of stone (Ming 2002).

Ancient Mining

Several kaolin deposits were located in the mountains in the Yaoli area, which includes Gaoling Mountain. The first sources were mined, like the china stone, by removing a layer of topsoil. Tunneling and open pit mining were also used. The deep-tunnel mines were triangularly shaped to take advantage of the natural rock strength. Where kaolin resources were plentiful, the cross sections of the tunnel were more irregular, and in mines with large spans, stone poles were used to stabilize the tunnels. The kaolin was brought out of the mountain as damp clumps and was processed near the mine to save costs. Processing required digging a trough into the side of the hill and constructing, on flat ground below, three brick-walled ponds connected by switch gates. The kaolin was first washed down the trough, and the fine kaolin particles overflowed into the first washing pond. There, the coarser particles of quartz, mica, and other impurities settled to the bottom and were held back by the walls as the gate was opened and the water/clay mix drained into the next pond. The overflow from pond to pond continued the purification until only the finest kaolin particles remained. In the last pond the clay was left to partially dry before being pounded into a wooden form to create a *dunzi*, or kaolin brick, weighing approximately two kilograms (kg) each. Similar bricks were made with china stone clay to allow for convenient formulation of the raw materials later, though the china stone was first crushed into a powder using a water-powered trip hammer. Gaoling Mountain workers carried the processed kaolin bricks in baskets seven kilometers down the mountain pathway to docks in the small village of Dongbu (FIG. 3B) along the Yao (East) River, from which it was taken by boat to Jingdezhen and mixed with china stone bricks in the desired proportions.

After He's *gaoling* discovery, news spread quickly and government officials confiscated the Macang mine, which had the best-quality kaolin. During the Yuan and Ming dynasties, this high-quality kaolin was used only for official wares, leaving the lesser-quality kaolin for civil kilns. This monopoly ensured the superiority of imperial ware. During the mid-Qing dynasty, Gaoling Mountain started supplying superior kaolin to both official and commercial kilns, which reenergized the porcelain production and allowed both imperial and commercial wares to flourish. Four large-scale mining sites were managed by four families—the Wang, the Feng, the He, and the Fang. The number-one pit on Gaoling Mountain began production in the Ming dynasty and lasted two hundred years. The largest mine measured 1000 meters in length and 50 meters in width and produced about 1,100,000 tons of kaolin. The Gaoling Mountain operation and other mines around Yaoli village were closed to further mining in 1965, and in 2008 the area was officially reopened as a National Mining Park.



FIGURE 4 The purest kaolin is called *gao bai ni*, or super-white clay, which has limited plasticity. Along with brilliance and whiteness, it can be trimmed to a thinness that allows sculpted walls to be uniquely translucent. This modern porcelain piece was produced from *gaoling tu*. *Yin Yang: Mitosis* by Gary Erickson, H. 20 cm × 38 cm × 28 cm, slipcast porcelain, slip decoration, 2013

Ancient Chinese Influences

The characteristics of Jingdezhen's refined white porcelain were highly sought by Chinese emperors. The first official agency, the Fouliang Porcelain Bureau, was established during the Mongol rule of the Yuan dynasty (1271–1368). At this time, the ceramic centers shifted from the northern to the southern part of China, bringing many experienced ceramic workers to Jingdezhen. The porcelain was mass produced by a very specialized and organized labor force. More than 500 masters and workers were divided among 21 to 23 departments, the most important being kiln masters, potters, painters, and writers of marks. There was also a host of other departments, including clay mixers and saggar makers (Emerson et al. 2000). Soon after, during the late Ming dynasty, there was a dramatic shift towards a market economy, and Jingdezhen began lucratively exporting to India, the Middle East, and Europe. The supply of resources and the location on the Changjiang, a river that connected to Poyang Lake and the Yangtze River, allowed easy and inexpensive transportation to major cities and seaports. At the height of production, there were over one thousand kilns in Jingdezhen.

As Europe had not yet discovered its own sources of kaolin, imported Chinese porcelain was highly admired for its whiteness, thinness, and exquisite painting. In the 17th century, its value exceeded that of gold as a commodity. It was collected only by the aristocracy, which even today feeds the perception that “fine China” is the most precious and expensive, though its production costs may be lower than those using other clays. The French Jesuit missionary priest Francois Xavier d'Entrecolles wrote two letters to his superiors in 1712 and 1722 in which he described Jingdezhen. His letters included detailed descriptions of porcelain-making techniques, which he learned through direct observation, through the help of his converts, and by consulting Chinese printed information. The letters were published in Europe and led to an increased interest in porcelain-making techniques. Soon,

the ceramic industry in Europe was growing by borrowing the Chinese techniques. The export business in China then saw a marked reduction (Emerson et al. 2000).

Contemporary Chinese Influences

Though Jingdezhen kaolin is historically important in the development of porcelain, the quality of its kaolin was actually very poor for ceramics as its alumina content was lower than in other kaolins. Gaoling Mountain kaolin is now only symbolically important because it was used in the first successful experiments of combining china stone and kaolin. Beginning in the Qing dynasty (1644–1911), kaolin began being shipped to Jingdezhen from many places in China. Contemporary kaolin mining sites are located in Fujian, Guangdong, Guangxi, Hebei, Hunan, Jiangsu, Yunnan, and Jiangxi provinces. The majority of kaolin used in Jingdezhen today comes from Fuzhou in Fujian province and from Xingzi town of Jiujiang city, located only 200 km away. Chinese and international ceramic artists still travel to Jingdezhen to utilize the special attributes of its super-white clay (Figs. 4, 5).

The allure of Chinese porcelain to Europeans promoted efforts to duplicate the production process in places like Cornwall, England. William Cookworthy discovered Cornwall kaolin deposits in 1745. However world-renowned Josiah Wedgwood had actually bought small consignments of kaolin from Georgia, USA, beforehand. It was not until Wedgwood acquired the ability to accurately control and measure kiln temperature that he founded his company in 1759 (Hudson 1969). Major exports from Georgia did not begin until the early 1960s. FIGURE 5 gives a timeline of changing pigmentation and firing technology, each change resulting in more vividly and intricately designed wares. Just as varied occurrences of kaolin and episodic economic deposits formed over Earth time, so have the art and sculpture of porcelain episodically changed with cultural time.



FIGURE 5 Eight ceramic wares depicting the dominance of Chinese influences and stylistic changes through time. (A) Chinese, Northern Song dynasty, late 11th–12th century, Ding ware. (B) Chinese, Song dynasty, 12th century, Jingdezhen ware. (C) Chinese, Yuan dynasty, 14th century, Jingdezhen ware. (D) Italian, Deruta, ca 1520–1525, maiolica, tin-glazed and cobalt blue. (E) Chinese, Ming dynasty, ca 1610–1620, Jingdezhen Kraak

blue underglaze. (F) Chinese, Qing dynasty, 1723–1735, Jingdezhen ware, cobalt blue underglaze and yellow enamel overglaze. (G) German, Meissen factory, ca 1725–1730, hard-paste porcelain with blue underglaze, enamel colors, and lustered. (H) English, modern ca 2011, Wedgwood earthenware. Details of plates in photos A–G (and photo credits) can be found in Emerson et al. (2000).

FUTURE PROSPECTS

The importance of kaolin through human history is intertwined with its place in Earth evolution, and this relationship will continue into the foreseeable future. A Web of Science search using the term *kaolin* for the past sixty years reveals a trend of 103, 204, 570, 871, 2176, 5974, and ~10,000 appearances, respectively, for ten-year increments up to the present. This exponential trend predicts >20,000 articles in the next decade. The fields of publication are also diversifying, from the geological and materials sciences to the environmental and medical sciences. In this issue, Balan et al. (2014) review the styles of kaolin-group layer-stacking defects and how this subject bears on industrial applications and the use of kaolins as environmental recorders. Kogel (2014) discusses concerns about energy efficiency, sustainability, and environmental awareness in the industry and shows how these concerns have led to advances in mining, processing, and reclamation practices. Cygan and Tazaki (2014) examine how kaolin minerals are being probed using computational chemistry and new spectroscopic tools to expand their applications

and to understand their significance in biology and our environment. Detellier and Schoonheydt (2014) note the crystallographic and elemental uniqueness of kaolin and explore how kaolin minerals can be nanocomposited to create material with novel properties. Finally, Williams and Hillier (2014) follow the curiosity of kaolin geophagy and its use in medicine, and they raise the following questions: what long-term health benefits and/or harm are associated with such practices, and what might kaolin offer in the development of new pharmaceuticals and disease treatment? The articles in this issue of *Elements* and the references therein indicate a bright future for kaolin.

ACKNOWLEDGMENTS

Gordon Brown was very helpful with the edits for all articles in the *Elements* kaolin issue. NSF, CRDF, IMERYs, Thiele, Active Minerals, KaMin, Kalemaden, UGA, and The Clay Minerals Society are all entities that have supported the research of PAS. Special thanks go to Hilary Schroeder for bringing the authors of this article together. ■

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