Halloysite mineral nanotubes – geology, properties and applied research

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Introduction

In 2010 seed funding through *PACE* was provided to commence a project that compared the properties of halloysite from a playa lake 60 km east of Maralinga, South Australia (the Camel Lake site), with halloysites from various geological settings within Australia, New Zealand and United States (Keeling 2010). Halloysite is a member of the kaolin group of clay minerals and typically crystallises as microtubules (Fig. 1). The objectives of the project were to raise awareness of geological factors that affect the physical properties of halloysite and to highlight what appeared to be special properties of the Camel Lake halloysite. The investigation has particular relevance to research into potential new applications in nanotechnology that seek to exploit the physical characteristics of halloysite as a natural mineral nanotube. While the extent of Camel Lake halloysite remains to be tested, samples taken from below thin sediment cover at the southwestern end of the dry lake bed have, to date, produced regular, straight halloysite tubules showing consistent range in length, tube wall thickness and diameter of the central cylindrical pore or lumen (Keeling, Self and Raven 2010).

Characterisation of halloysites was done at Adelaide University and CSIRO Land and Water as a post doctorial study by Pasbakhsh under the guidance of Professor David Chittleborough and Dr G Jock Churchman. Preliminary data was presented in 2011 at the International Congress for Applied

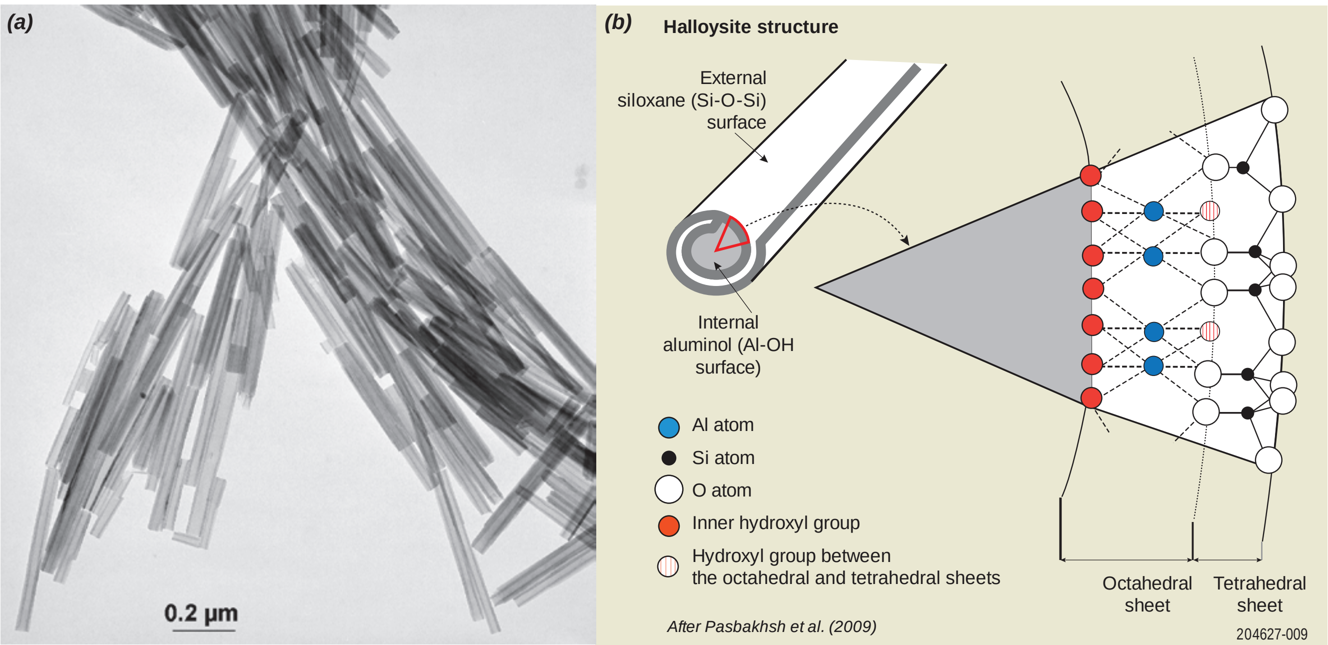

Figure 1 (a) Straight, regular halloysite microtubles from Camel Lake. (b) Halloysite crystal morphology and atomic structure. (Photo 414524)

Mineralogy held at Trondheim, Norway (Keeling, Pasbakhsh and Churchman 2012), with full results published subsequently in *Applied Clay Science* (Pasbakhsh, Churchman and Keeling 2013). The release of data saw an increase in requests from researchers for samples of Camel Lake halloysite, which has been met from limited stocks held by the Geological Survey of South Australia. The continuing growth in research interest in halloysite prompted an approach from Apple Academic Press Inc. regarding the possibility of a book on the topic of 'Natural mineral nanotubes: properties and applications'. Participation was sought from key researchers and clay mineral experts, and the assembled work was published in January 2015. The book includes an overview of the mineralogy and chemistry of key mineral nanotubes: chrysotile, halloysite, imogolite, allophane, palygorskite and sepiolite (Guggenheim 2015), together with an outline of their geological occurrences including a chapter on halloysite by Keeling (2015). Over two-thirds of the book content is given to reviews and descriptions of recent research activity on modification of mineral nanotubes to enhance their use, principally as functional fibre reinforcement, as nanocontainers, and as fibre templates for improved materials for filtration and catalysis (Pasbakhsh and Churchman 2015). The results of the initial *PACE*-funded project and some of the outcomes that developed from this work, particularly in relation to understanding the conditions of formation of Camel Lake halloysite, its properties and potential uses, are summarised below.

Geology and formation of halloysite

Halloysite is a relatively common mineral that often crystallises together with kaolinite; the two polymorphs of kaolin are not easily separated. Deposits suitable for commercial development are, however, comparatively rare and occur either as relatively pure masses of halloysite (e.g. Dragon Mine, Utah; Camel Lake) or as large, lower-grade sources from which halloysite can be readily separated (e.g. Matauri Bay deposits, New Zealand). Geological environments include volcanic rocks altered by supergene or low-temperature hydrothermal fluids, or where acid groundwater interacts with reactive silicates, often in close proximity to carbonate rocks. Thermodynamic properties of kaolinite favour its formation over halloysite and this is reflected in the far greater relative abundance of kaolinite. Halloysite, however, has lower activation energy of nucleation than kaolinite and would be expected to be the first kaolin mineral to precipitate from solution (Steefel and van Cappellen 1990). This situation is enhanced where pre-existing silicates undergo rapid leaching and dissolution and where few mineral substrates are available for nucleation of kaolinite crystallites. Formation and preservation of halloysite is more likely in an environment that remains wet but where fluid chemistry fluctuates, in particular, where cycles of corrosive (acidic) fluids interact with volcanic glass or less stable silicate minerals such as Ca-plagioclase or smectitic clays under the influence of a steep chemical or thermal gradient that modifies the solubility of Al and Si ions, leading to rapid nucleation of halloysite crystallites. Under these conditions, fluid saturation levels are quickly lowered to the point where any kaolinite precursors are resorbed and the predominance of halloysite is enhanced (Fritz and Noguera 2009).

These conditions are reflected in the formation of the original 'type' halloysite described and named by Berthier (1826) from Angleur near Liége, Belgium. At Angleur, waxy white halloysite clay was found in karstic cavities in Lower Carboniferous limestone in association with Pb and Zn sulfides, and the products of sulfide weathering. The large, open karstic features in Carboniferous limestone forming the Entre-Sambre-et-Meuse plateau in Belgium were mostly infilled by Cenozoic terrestrial sandy sediment, containing organic matter and pyrite, and interbeds of lignitic clay (Fig. 2). Dupuis et al. (2003) argued that meteoric water passing through these sediments progressively oxidised pyrite and

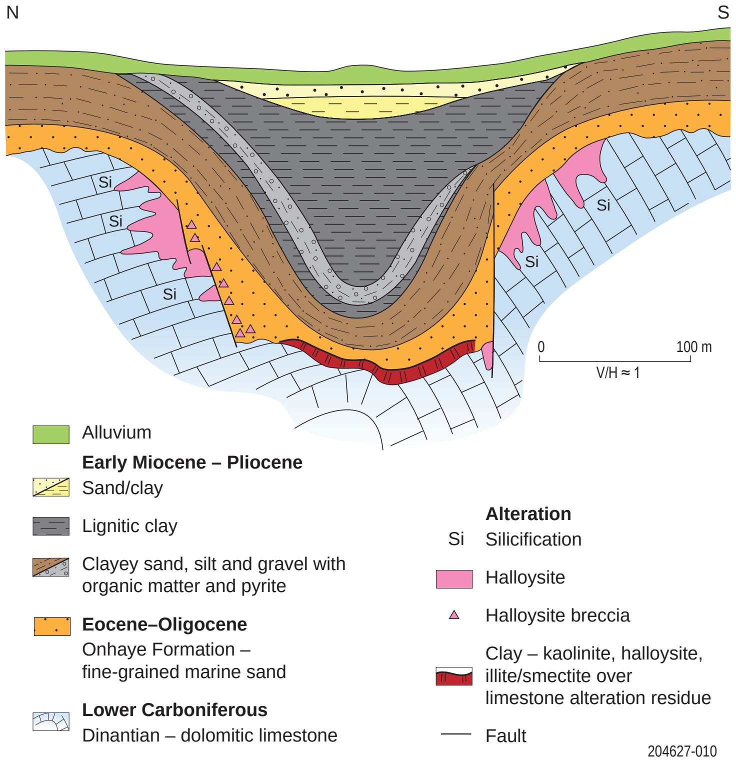

Figure 2 *Halloysite deposit setting, Entre-Sambre-et-Meuse plateau in Belgium, formed in crypto-karstic dolomitic limestone by acid groundwater from sediments infilling the karst. (Modified from Dupuis et al. 2003)*

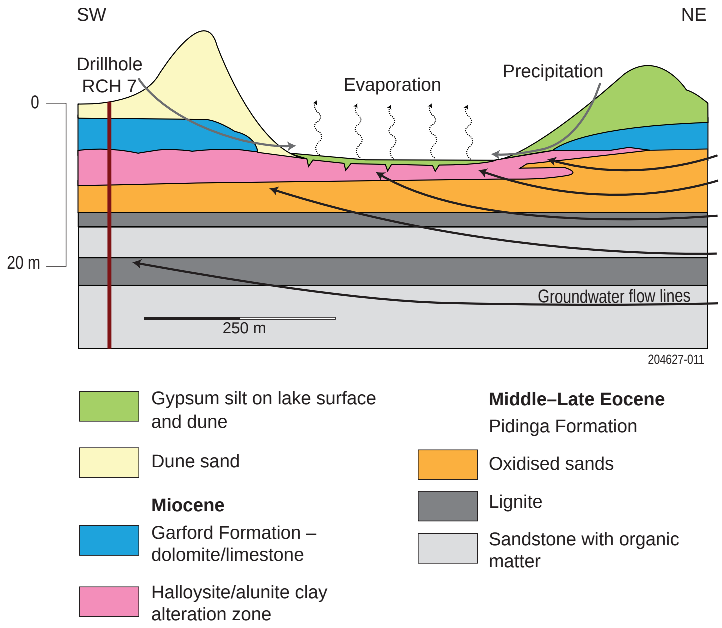

Figure 3 *Geological setting of halloysite deposits formed by acid groundwater at the Camel Lake site, near Maralinga, South Australia.*

acidified the groundwater. This led to dissolution of silicates within the more permeable sandy layers and increased the levels of Al and Si ions in solution. Precipitation of aluminium, as gibbsite or halloysite, was at the interface with the limestone where the solution pH was modified and the solubility of aluminium reduced; silica activity was maintained by acid groundwater flow in the leached quartz-rich sediment. Kinetic chemical reaction modelling by Dupuis et al. (2003) indicated that early formed gibbsite would subsequently have recrystallised to halloysite and excess silica precipitated along fluid pathways within the limestone, which accorded with the pervasive occurrence of silicified limestone in the rim of the karst.

A similar situation is envisaged in the formation of Camel Lake halloysite. In this case, acidic saline groundwater was developed during oxidation of lignitic, pyritic sands of Eocene Pidinga Formation within buried fluvial channels that drained towards the coastal margin of the former marine Eucla Basin. Uplift through regional, gentle warping (Hou et al. 2008) and drier climatic conditions in the Late Neogene both contributed to a general lowering of groundwater levels that likely accelerated oxidation of reduced sediments in the paleovalleys. In the region of Camel Lake, lateral groundwater flow within the channels was, and continues to be, towards the southwest and west, with discharge zones in low-lying areas behind former offshore barrier islands and paleocoastal dunes that mark the landward extent of the Eucla Basin. Intermittent groundwater discharge continues to occur in eroded hollows where Eocene sands are exposed in present-day playa lakes. At these sites, evaporation results in increased salinity and acidity leading to precipitation of gypsum and alunite. The Pidinga Formation sands are extensively overlain by Miocene lacustrine illite/smectite clays grading upwards into dolomite and pisolitic limestone, collectively termed Garford Formation. The thin clay and carbonate units buffer groundwater acidity at discharge sites. This is the situation at Camel Lake where acid groundwater from oxidised Eocene sands interacts with overlying Garford Formation (Fig. 3). A chemical gradient is established that likely controlled, over an extended period, the dissolution of illite/smectite clays and crystallisation of halloysite (Fig. 4), which is up to 4 m thick at the contact with Garford Formation dolomite (Keeling, Self and Raven 2010).

Comparison of halloysite characteristics

Halloysite from Camel Lake was examined and compared with five halloysites from various locations in Australia, New Zealand and United States. These included commercial halloysite product from Matauri Bay, Northland, New Zealand (weathered late Cenozoic alkaline rhyolite; Brathwaite et al. 2012) and the Dragon Mine, Utah, United States (Paleogene hydrothermal alteration and replacement of Cambrian dolomite; Kildale and Thomas 1957).

Figure 4 *Collecting halloysite samples (site CLA-1) from below thin sediment cover at the Camel Lake playa. Halloysite makes up between 35 to 72% of the clay alteration. (Photo 414534)*

Data were obtained for major oxide chemistry by X-ray fluorescence, mineralogy by powder X-ray diffraction, composition of exchangeable cations, particle morphology and dimensions from multiple transmission electron micrographs, surface area and porosity by nitrogen adsorption, and surface charge characteristics by measuring the change in zeta potential over pH range 1.5–12 (Pasbakhsh, Churchman and Keeling 2013). Selected results for Matauri Bay halloysite (MB-NN; upgraded commercial product supplied by Natural Nano Inc.), Dragon Mine halloysite (DG; product supplied by Applied Minerals Inc.) and Camel Lake halloysite (CLA; washed, decanted and dried sample from site CLA-1) are summarised in Tables 1–3.

All three samples were comprised of dominantly tubular forms of halloysite, variously estimated at 97% (MB-NN), 84% (DG) and 95% (CLA). The lower halloysite content in sample DG was due to the presence of kaolinite (8%), gibbsite (3%), quartz (3%) and alunite/woodhouseite (1%). Sample CLA also contained alunite (2.7%) with traces of quartz (1%) and iron oxide (1%), while sample MB-NN contained traces of probable cristobalite (1%) and feldspar (1%). A high proportion of thicker and longer tubes in MB-NN produced comparatively low values for surface area (29.3 m^2/g), pore volume (0.077 cm^3/g) and overall proportion of lumen pore space (16.3%). This contrasted with sample CLA with total surface area 74.6 m^2/g, pore volume 0.17 cm^3/g and a high percentage of lumen pore space of 33.8%. Sample DG had a high proportion of small short tubes which were responsible for the relatively high surface area of 57.3 m^2/g and lumen pore space of 26.3%.

Properties of fine particle size and low iron oxide and titania contents are key requirements for traditional halloysite markets in high quality ceramic manufacture. For potential new applications as functional fillers and nanocapsules, however, particle size, shape, pore volume and pore dimensions are more likely to influence the performance of products under investigation. Thicker tubes with a wide range of lumen capacity may offer advantages for particular applications, whereas regular tubes with high surface area and high pore volume offer improved dispersion and bonding, with high internal loading capacity. While significant recent progress has been made with incorporation of halloysite in polymer systems to improve tensile strength and impact resistance, other promising areas of research, particularly as nanocontainers and in medical applications, are at early stages of development with no clear pathway for high volume or high value commercial uptake. This field of applied research is very dynamic with potential applications across diverse industries including defence, cosmetics, healthcare, agriculture, environment, catalysts and polymer manufacture (e.g. Lvov et al. 2008). A few examples of recent research outcomes are described below, some of which involve comparisons with halloysite from the Camel Lake site.

Table 1 Halloysite morphological characteristics from transient electromagnetic images

Halloysite	Particle shapes	Length (nm)	Inner diameter (nm)	Outer diameter (nm)	Wall thickness (nm)	Aspect ratio (average)
Matauri Bay, New Zealand (MB-NN)	Tubular: long, thin or thick with some short and stubby. Minor spheroidal and platy forms.	100–3000	10–35	40–150	10–100	16:1
Dragon Mine, Utah (DG)	Tubular: short, thin and stubby tubes with some long and thin. Minor platy kaolinite.	50–1500	5–30	20–150	5–50	9:1
Camel Lake, South Australia (CLA)	Tubular: regular, thin and of various length.	100–1500	10–50	20–70	5–30	17:1

Table 2 Halloysite surface area and average pore size properties (from Pasbakhsh, Churchman and Keeling 2013)

Halloysite	S_{BET} (m^2/g)	S_{abs} for pores 1.7–300 nm diameter (m^2/g)	V_{abs} for pores 1.7–300 nm diameter (cm^3/g)	Average pore size (nm)	V_{abs} for pores in the range of lumen diameter (cm^3/g)	Lumen space (%)
MB-NN	29.3	30.5	0.07	10.0	0.06	16.3
DG	57.3	58.8	0.12	8.1	0.10	26.3
CLA	74.6	76.6	0.17	8.9	0.13	33.8

S_{BET} specific surface area; S_{abs} specific surface area from adsorption isotherm; V_{abs} BJH adsorption cumulative volume of pores.

Table 3 Halloysite major oxides (from Pasbakhsh, Churchman and Keeling 2013; Keeling 2015)

Halloysite	SiO_2 (%)	TiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	MnO (%)	MgO (%)	CaO (%)	Na_2O (%)	K_2O (%)	P_2O_5 (%)	SO_3 (%)	Sum (%)	LOI (%)
MB-NN	45.82	0.12	38.21	0.48	0.01	0.17	0.01	0.12	0.01	0.32	0.02	85.30	14.7
DG	43.50	0.02	38.88	0.33	0.001	0.12	0.26	0.07	0.07	0.83	0.26	84.34	15.7
CLA	44.96	0.15	37.57	1.21	0.01	0.19	0.28	0.09	0.31	0.01	0.63	85.47	14.5

LOI loss on ignition.

Applied research incorporating Camel Lake halloysite

Halloysite dispersion in low concentration in polymer systems has been shown to be relatively straightforward, with or without pre-modification of the halloysite surface, and can produce significant improvement in mechanical and thermal performance when incorporated in polypropylene (PP), ethylene propylene diene monomer (EPDM), styrene-butadiene rubber and epoxy (Vahedi and Pasbakhsh 2015, and references therein). In testing improvement in tensile strength of EPDM incorporating halloysite nanotubes (HNTs), Pasbakhsh et al. (2014) compared halloysites from Matauri Bay (MB), Jarrahdale (Western Australia) (JA), and Camel Lake (CLA). EPDM incorporating CLA outperformed MB and JA nanocomposites at all concentrations, producing a maximum 340% increase in tensile properties with halloysite loading of 15% phr (parts per hundred of rubber; Fig. 5a). The difference in performance was attributed by the authors to the combination of higher aspect ratio and higher surface area of CLA compared with the other halloysite nanotubes. Similar high performance was recorded for CLA in tests on improved impact properties for epoxy/halloysite nanocomposites (Vahedi, Pasbakhsh and Chai 2015; Fig. 5b).

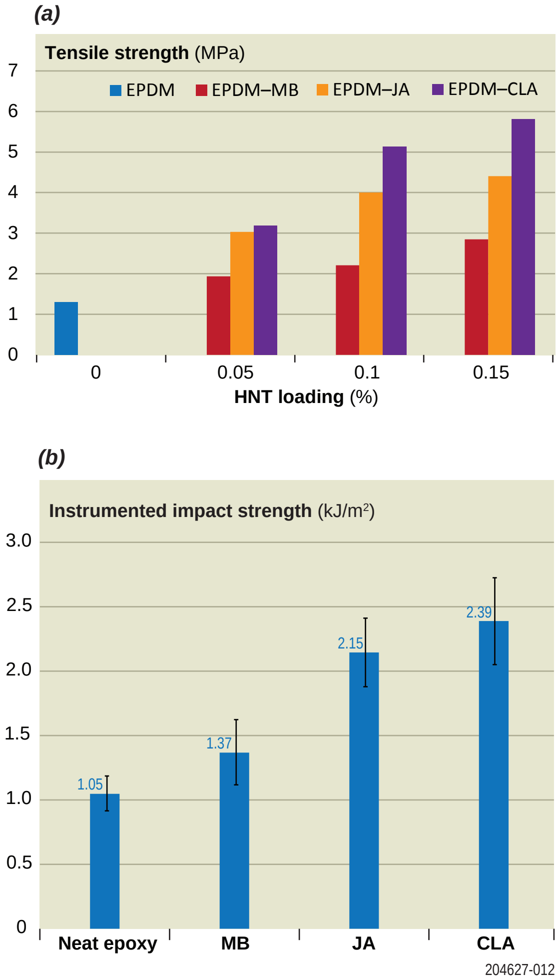

Figure 5 *Influence of halloysite type on (a) tensile strength of synthetic rubber (EPDM) and (b) impact resistance for epoxy/halloysite nanotubes composites. MB (Matauri Bay), JA (Jarrahdale), CLA (Camel Lake). (From Pasbakhsh et al. 2014; Vahedi, Pasbakhsh and Chai 2015)*

Halloysite has been exploited over many decades without evidence of toxicity to humans; an observation supported by recent animal and cell viability studies, summarised in Abdullayev (2015). The results attest to the mineral's general biocompatibility and low environmental impact. Investigations of Camel Lake halloysite, sample CLA-1, during 2011 at the Universidad Autónoma Metropolitana, Mexico, reported the first observation of anti-inflammatory properties of halloysite, comparable to that of indomethacin (Cornejo-Garrido et al. 2012). Evidence was based on mieloperoxidase enzymatic activity, using adult male Wister rats, and interaction with mice primary peritoneal macrophages, which showed that CLA-1 inhibited the production of nitric oxide and inhibited oxidative stress via lipid peroxidation. Subsequent studies on halloysites from various locations showed anti-inflammatory activity and cytotoxcicity varied between samples and was correlated with surface area; highest anti-inflammatory activity was associated with halloysites of high surface area (Cervini-Silva, Nieto-Camacho and Ramírez-Apan 2015).

Recent research developments – selected examples

Halloysite is not biodegradable but has been investigated for a variety of healthcare and cosmetic applications, including external wound care, bone implants and dental fillers. Halloysite nanotubes have been shown to be effective for encapsulation and sustained release of drugs and an approach to control the timing of release has been developed by coating the tubes with polyelectrolyte (e.g. chitosan, gelatine; polyvinylpyrrolidone, PVP; poly-acrylic acid, PAA), using layer-by-layer assembly to give multilayer shells of required thickness (Veerabadran et al. 2009). Average lumen diameters of 8–10 nm allow halloysite to accommodate macromolecules within the lumen. Also, the hydroxyl groups on the internal aluminol surface of the halloysite tubes can be selectively modified to enhance uptake of the guest drug (Tan et al. 2013). Modification of the external siloxane surface of halloysite by surfactant sodium dodecanote was reported by Mitchell, Castellanos and King (2015) as an effective method for selective adhesion of circulating tumour cells to facilitate their collection from blood. The study involved coating the internal surface of 300 µm diameter microenathane tubing with modified halloysite, which provided surface roughness and a high contact area. Effectiveness and selectivity were determined by identifying and measuring attached cells following controlled infusions of mixtures of cancer cells and leukocytes. The spread of cancer cells from a primary tumour via blood circulation is a major cause of cancer-related deaths. Concentration and collection of circulating tumour cells has application in cancer diagnosis and treatment aimed at preventing formation of secondary tumours.

Unmodified halloysite is environmentally benign, but the incorporation of halloysite with graphene and nanoparticulate silver by researchers at Zhengzhou University, China, created an enhanced antibacterial reagent, shown to be highly effective against resilient Gram-positive bacteria (*Staphylococcus aureus*) and Gram-negative bacteria (*Escherichia coli*; Yu et al. 2014). The thin edges of graphene oxide (GO) stress and fatally damage bacteria cell membrane; the effect is reduced where the GO nanosheets are aggregated. Silver nanoparticles block functional operations of micro-organisms to give broad-spectrum antibacterial action and some antiviral activity, yet are non-toxic to the human body at low concentrations. The silver nanoparticles, however, readily agglomerate, which decreases the antibacterial effect. The function of halloysite was to locate between the graphene oxide sheets to limit the effects of aggregation and to provide high surface area for deposition of silver nanoparticles. This was achieved by coating the halloysite nanotubes with organic adhesive dopamine and dispersion with GO nanosheets in Tollens' reagent (silver nitrate/ammonia mix; Fig. 6). In the process graphene oxide was partly reduced to graphene (rGO), with lower tendency to aggregate. Silver nanoparticles (5–15 nm across) were formed via reduction and deposited on the surfaces of graphene and halloysite with minimal agglomeration. The resulting sandwich-like silver nanoparticles/halloysite nanotubes/graphene nanocomposite (Ag/HNTs/rGO), with high surface area of 278m^2/g, could be dispersed at low concentration to provide a highly effective biocide or be incorporated in polymer film with potential application as antibacterial membrane for water treatment (Yu et al. 2014).

Innovative developments, such as those outlined above, provide encouragement for future growth in demand for specialist materials based on new 'nano minerals' such as halloysite and graphene, both of which could be readily obtained from natural resources in South Australia.

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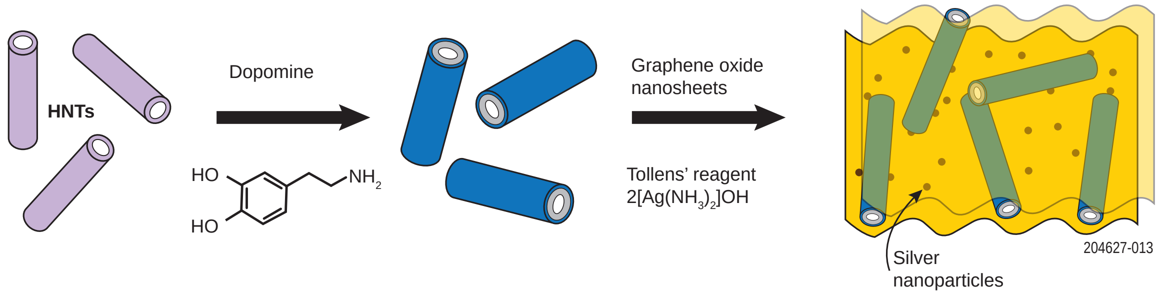

Figure 6 *Fabrication process for silver nanoparticles on halloysite nanotube/graphene composites (Ag/HNTs/rGO). (Adapted from Yu et al. 2014)*

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FURTHER INFORMATION

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Roger Goldsworthy AO – supporting South Australia's resources sector

On Wednesday 30 September 2015 the Treasurer of South Australia, Hon Tom Koutsantonis MP, hosted a thank you dinner at the South Australian Museum for the Hon Eric Roger Goldsworthy AO. Roger, as he prefers to be known, has been a champion of the South Australian resources sector since he entered politics in 1970. He held key parliamentary positions such as deputy leader of the opposition (Liberal Party, SA); deputy premier; minister of mines and energy, services and supply; and chairman of the Budget Review Committee. As minister, Roger's most well-known achievements included: negotiations to establish the Olympic Dam copper–uranium mine and introduction of the Roxby Downs Indenture Bill to parliament on 5 March 1982. The *Roxby Downs (Indenture Ratification) Act 1982* came into operation on 21 June 1982. He was also key in the negotiations of the Cooper Basin Liquids Project Indenture and involved in Pitjantjatjara land rights negotiations. Roger was awarded the Officer of the Order of Australia for service to politics, to the parliament of South Australia and to the community in 1997, and a Centenary Medal in 2001 for his service to the South Australian Parliament as a minister and deputy premier. After retiring from politics in 1992 Roger was kept busy with various roles on boards and committees. With Roger's recent retirement from the Resources Industry Development Board (now known as the Minerals and Energy Advisory Council) the celebratory dinner was a timely reminder of his achievements. ■

(Photos 414651–414653)