# The Development of Nano-Sericite Process

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### **ABSTRACT**

In the Shiangyang region of Taitung County, Taiwan, there is a rich lode of sericite which is one of the most economically valuable minerals in Taiwan. Because in between the plate layers of the mineral, there are potassium ionic bonds exfoliation of the mineral is often difficult. If the potassium content could be reduced or substituted with other ions to decrease strength of the ionic bonds, then the mineral might be more easily intercalated or exfoliated. The purpose of this study was to use the molten salt intercalation method to achieve intercalation or exfoliation of sericite and thus allow diminution of the mineral particles as nano-scale raw material. The experiments entailed using an oil bath to heat varying amounts of sodium hydroxide (at 0, 10, 20, 30, and 45 g to 5 g sericite) or oxalic acid (at 0, 2, 5, 8, 10 g to 5 g sericite) to temperatures between 100 and 200 and observe the ensuing displacement or intercalation efficacy on the sericite layers. The treated sericite was then swelled using nitric acid neutralization. The before and after treatment sericite specimens were examined using an X-ray diffractogaphy (XRD), scanning electron microscopy (SEM), and its energy dispersive spectroscopy (EDS) attachment to observe whether the lattice spacing occurred. The specific surface area of the modified sericite was assessed by the Brunauer-Emmett-Teller (BET) and methylene blue adsorption methods. The post-treatment solution was also analyzed with an inductively coupled plasma (ICP) analyzer to see whether potassium ion was substituted. The XRD results indicate that simply heating sericite raw material to 100, 150 and 200 caused no modification of the crystal lattice spacing. There was no discernible structural modification by SEM examination as well. With the addition of sodium hydroxide, the XRD results suggest that diffraction peaks corresponding to impurity pyrophyllite diminished with increasing sodium hydroxide dosage and temperature increases. At the 20 g NaOH dosage and 200 , sericite crystal lattice spacing started to change. The 45 g treated and acidified specimen showed diffraction peaks shifting from the original 4.36 and 6.43 A to 4.36 and 7.90 A, with marked increase in the interlayer spacing. At the condition of 200 , the specific surface area of the sericite increased with the NaOH dosage from the original 23.7 m<sup>2</sup>/g to 78.0 m<sup>2</sup>/g at the 45 g NaOH dosage. The results of sericite lattice spacing and specific surface area, heating by oil bath with NaOH lowered crystallinity of the mineral and expanded interlayer spacing. The cationic exchange capacity (CEC) increased from the original 4.98 meg/ 100 g to 46.8 meg/100 g at the 45 g NaOH dosage. With the addition of oxalic acid, on the other hand, the XRD results showed nearly identical diffraction patterns with similar intensities, indicating a lack of exfoliation or modification of interlayer spacing. The specific surface area even decreased with the addition of oxalic acid. Thus, oxalic acid was deemed ineffectual in modification of sericite.

Keywords: sericite; molten salt intercalation method; nano-scale; nano-scale exfoliation nano-scale exfoliation exfoliation; methylene blue

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#### REFERENCES

1.石大鑫,1992,中國大陸之雲母與石綿,礦業技術,中國大陸,30:2-19。2.李宗銘,1998,半導體封裝材料發展趨勢,工業材料 ,139:108-116。 3.岩生周一,1967,黏土,技報堂出版,日本東京。 4.吳景雅,2000,向陽絹雲母之離子性質交換研究,碩士論文,國 立成功大學礦冶及材料科學系。 5.周錦煜,2001,絹雲母之超細粉碎研究,碩士論文,國立台北科技大學材料及資源工程系。 6.武英傑 ,2003,導電雲母奈米複合材料之製備,碩士論文,國立東華大學材料科學與工程系。 7.施育仁,2003,以水熱程序進行向陽絹雲母剝 層改質之研究,碩士論文,國立成功大學資源工程學系。 8.馬振基,2003年3月,奈米材料科技原理與應用,全華科技圖書,台北市。 9.郭魁士,1997,土讓學,中國書局,台北縣。10.陳仕宏及雷大同,2001年12月,向陽絹雲母之粒徑縮減研究,資源與環境研討會論 文集,大漢技術學院,花蓮。 11.逢甲大學奈米科技研究中心,2002, http://www.nano.fcu.edu.tw/nm.scopes.html 12.森田修二,1962, 土壤學汎論,養賢堂,日本東京。 13.張志誠, 2002, 奈米技術全面報到, 就業情報, 319:42-54。 14.張立德, 牟季美, 2002年6月, 奈 米材料和奈米結構,滄海書局,台中市。 15.張立德,2003年2月,第四次浪潮-奈米衝擊波,中國經濟出版社,北京。 16.張振輝,2005 ,向陽絹雲母礦中絹雲母及葉臘石水熱改質研究,碩士論文,國立成功大學資源工程學系。 17.經濟部礦業司,1995,台灣地區雲母之 利用需與流向調查,台灣礦業,47(4):451-467。 18.經濟部礦業司,1995,向陽絹雲母專業區規劃開發計畫,經濟部委託研究報告。 19. 溫紹炳,雷大同,黃紀嚴,2000,台灣片狀矽酸鹽礦之利用開發計畫報告,經濟部礦業司。20.趙杏媛、張有瑜,1990,黏土礦物與黏 土礦物分析,海洋出版社,北京。 21.葉瑞銘,2004,奈米科技導論,高立圖書出版,台北縣。 22.蔡宗燕,1998,奈米級無機材料的發 展與應用,化工資訊,1998(2):28-42。 23.劉玉梅及雷大同,2003,向陽絹雲母晶格離子置換研究,資源與環境學術研討會,主辦單位, 花蓮。 24.盧希鵬,馬振基,2003,奈米材料技術地圖,國科會科學技術資料中心。 25.賴仁堯,2005,奈米級絹雲母顏料之研製與其塗 布紙之應用,碩士論文,國立屏東科技大學木材工業系。26.蕭朝光,2003,向陽絹雲母粒徑縮減及晶格離子置換研究,碩士論文,國 立成功大學資源工程學系。 27.譚立平及魏稽生編著,1997,台灣非金屬經濟礦物,經濟部中央地質調查所。 28. Alexandre M, Dubois P. 2000. Polymer-layered silicate nanocomposites: preparation, properties and uses of a new class of materials. Mater Sci Eng 28:1-63. 29. Becker M, Schwedes J. 1999. Commiuntion of ceramics in stirred media mills and wear of grinding beads. Powder Technol 105:374-381. 30. Caseri WR, Shelden RA, Suter UW. 1992. Preparation of muscovite with ultrahigh specific surface area by chemical cleavage. Colloid Polym Sci 270: 292-298. 31. Cho JW, Paul DJ. 2001. Nylon 6 nanocomposites by melt compounding. Polymer 42: 1083-1094. 32. Cho H, Waters MA, Hogg R. 1996. Investigation of the grind limit in stirred-media milling. Int J Miner Process 44-45:607-615. 33. Ikazaki F, Uchida K, Kamiya K, Kawai A, Gotoh A, Akiba E. 1996. Chemically assisted dry comminution of sericite- dry comminution method accompanied by ion-exchange. Int J Miner Process 44-45:93-100. 34. Kojima Y, Usuki A, Kawasumi M, Okada A, Kurauchi T, Kamigaito O. 1993. Sorption of water in nylon 6-clay hybrid. J Appl Polym Sci 49:1259-1264. 35. Osman MA, Christoph M, Walter R, Ulrich W. 1999. Alkali metals ion exchange on muscovite mica. J Colloid Interface Sci 209:232-239, 36, Rajamani K, Songfack P, Mishra BK, 2000, Impact energy spectra of tumbling mills, Powder Technol 108(2-3):116-121. 37. Tomita K and Toshio S. 1971. Transformation of sericite into an interstratified mineral. Clays Clay Miner 19:263-270. 38. Tomita K. 1977. Experimental transformation of 2M sericite into a rectorite-type mixed-layer mineral by treatment with various salts. Clays Clay Miner 25: 302-308. 39. Usuki A, Kojima Y, Kawasumi M, Okada A, Fukushima Y, Kurauchi T, Kamigaito O. 1993. Synthesis of nylon 6-clay hybrid. J Mater Res 8(5):1179-1184.