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1 Research paper

Q2 How the unique properties of soil kaolin affect the fertility of tropical soils

Q3 Robert J. Gilkes ^{a,*}, Nattaporn Prakongkep ^b

^a School of Earth and Environment, University of Western Australia, Crawley 6009, Australia

5 b Soil Mineralogy and Soil Micromorphology Research Group, Office of Science for Land Development, Land Development Department, Bangkok, Thailand

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34 1. Introduction

Should a paper on the significance of soil kaolin (s.l.) be included in a 35 36publication that is mostly concerned with commercial clay minerals? 37Our response is simply that kaolin dominated soils are abundant in the wet tropics and as a high proportion of the world's population relies 38 on these soils for the agriculture that produces food and fiber (Fig. 1) we 39 should be aware of these kaolins. Kaolin enables these soils to provide 40clean water and buffer the environment against diverse contaminants. 41 To the many people in this region the unique properties of soil kaolin 42 are of critical importance so that the mineral deserves the research at-43 tention that it has received in recent decades. We have chosen to use 44 45 the term kaolin rather than kaolinite as soil kaolins may include minerals that might be classified as variously ordered kaolinites, hallovsite. 46nacrite and dickite but are not readily distinguished by routine exami-47 nation. We are therefore considering the kaolin group of minerals as 4849 discussed by Guggenheim et al. (1997) but also include halloysite in its variously hydrated conditions. 50

Soil kaolins are as diverse as those used in industry but they may dif-5152fer from industrial kaolins in several important respects. The kaolin present in the solum (the uppermost "living" part of the soil) is com-53 monly different from kaolin in the underlying saprolite or sediment 5455where the kaolin may resemble many industrial kaolins; consisting of large, structurally ordered, euhedral crystals (Varajão et al., 2001; 5657Hughes et al., 2009). The specimen kaolins described in many publica-58tions commonly consist of large, euhedral and highly ordered crystals 59 (Hart et al., 2002; Hughes et al., 2009). Soil kaolin crystals are mostly

* Corresponding author. E-mail address: bob.gilkes@uwa.edu.au (R.J. Gilkes).

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ABSTRACT

The kaolin (*s.l.*) in many tropical soils is distinctly different from most specimen and industrial kaolins. Crystal 14 structure is highly disordered and crystal sizes are much smaller than for specimen and industrial kaolins. The 15 crystal habit of soil kaolins is diverse ranging through euhedral hexagonal and anhedral platy, spheroidal and tubular. The specific surface of soil kaolins is higher than for most industrial kaolins. Consequently, cation and anion 17 retention capacities are higher although there is no compelling evidence that the high structural disorder of soil 18 kaolin is associated with a greater specific reactivity of the kaolin surface. A small amount of ferric iron substitutes 19 for octahedral Al but there is generally little or no substitution of other cations. Soil kaolin commonly contains 20 minor amounts of potassium but this is present in mica layers within, attached or associated with kaolin crystals 21 so that potassium is not within the kaolin structure. Due to its considerable chemical reactivity soil kaolin helps 22 provide an effective substrate to support agriculture and other land uses in the tropics. 23

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very small with habits ranging from euhedral hexagonal plates through 60 anhedral plates to tubular and spherical forms. Consequently, specific 61 surface area (SSA) is high resulting in substantial cation and anion re- 62 tention capacities which are of great benefit to agriculture as cationic 63 and anionic forms of plant nutrient elements are retained against 64 leaching under the high rainfall condition of the wet tropics. Similarly 65 soil kaolin can reduce the mobility of toxic ions and harmful synthetic 66 organic molecules in soils thereby moderating contamination of food 67 crops and water resources. An important and desirable property of soil 68 is its capacity to retain the water received from rainfall or irrigation 69 and then release this water to plants in response to the demands of 70 the growing plant transmitted through the root system. What is re-71 quired is a stable, porous soil structure with an abundance of connected 72 pores in the 0.1-10 µm size range. Many tropical soils with kaolin as the 73 dominant clay mineral have exactly this characteristic and consequently 74 they are less sensitive to drought conditions and water is used efficient-75 ly. The low bulk density and structural stability of some kaolin-rich trop-76 ical soils, notably oxisols, are thought to be a consequence of their stable 77 low-density, porous "card house" structure where kaolin plates are se-78 cured in place by iron oxide crystals attached to kaolin crystals through 79 electrostatic attraction (Tawornpruek et al., 2006). Kaolin has a negative 80 surface charge and iron oxides have a positive surface charge at the acid 81 pH conditions prevalent in tropical soils (Khawmee et al., 2013). 82

A commonly held view of the mineralogical basis of chemical soil 83 fertility is that the clay fraction contains micrometric grain of mica, ver-84 miculite, chlorite, feldspar, pyroxene, and other primary minerals 85 (Retallack, 1990). These minerals contain several plant nutrient ele-86 ments that are released to plants by dissolution and exchange reactions 87 in the soil. This is certainly the situation in mid-latitudes where many 88 soils have formed on glacial residuum which includes alluvium and 89

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Fig. 1. (a) The global distribution of kaolinitic soils (based on ISSS, 1998) and (b) The global distribution of medium to high population density showing that the highly populated tropical region commonly has kaolin-rich soils (based on CIESIN-Columbia University, 2014).

loess transported for long distances by wind and water from regions of 90 former glacial activity. Indeed some tropical soils have also been derived 91 92from rock debris resulting from tectonic activity and vulcanicity and 93 these soils are consequently fertile. Large active river systems in the tro-94pics deposit mineral rich alluvium in flood plains and deltas which sup-95port fertile productive soils (Young, 1976). However, much of the 96 tropical and subtropical zone is occupied by ancient upland landscapes 97 mantled by deep, highly weathered regolith with kaolin and iron oxides dominating the clay fraction of the solum. Quartz and a few resistant 98 primary minerals comprise the silt and sand fractions which do not con-99 tribute significant amounts of nutrients to plants (Darunsontaya et al., 100 2012). Under these circumstances, it might be anticipated that the 101 102chemical fertility of the prevalent kaolinitic soils will be poor and this is usually the case. Soil organic matter may provide a reserve of some 103 plant nutrients and is of particular value in highly weathered kaolinitic 104 105 soils but the soils generally remain infertile. There is evidence for potassium and other plant nutrient elements being present in minor concen-106 107 trations in soil kaolin and these elements may be at least partly available to plants (Singh, 1991). 108

It is evident from this overview that the properties of soil kaolins are 109rather different from specimen kaolinite and those kaolins utilized by 110 industry and also that the properties of kaolin that are of particular in-111 terest to soil scientists may not coincide with those of researchers into 112 the properties and performance of industrial and specimen clays. In 113 this paper, we review the present state of knowledge of soil kaolin 114 and attempt to identify relationships between chemical, morphological 115116 and structural properties of soil kaolins. We restrict our review to those papers that have used the same analytical procedures and we have been 117 alert to data having been reproduced in several papers thus avoiding 118 duplication of data in our summaries of experimental results. 119

120 2. Preparing soil kaolin for analysis

121 It is impossible to separate pure kaolin samples from soils as the clay 122 fraction of all soils contains micrometric crystals of diverse accessory 123 minerals. Consequently, measurements are made on clay separates in-124 cluding clays that have been chemically treated to remove Fe/Mn ox-125 ides, carbonate minerals and organic matter, but these separates are still not pure kaolin. This is a particular problem for measurements of 126 surface properties of soil kaolin (SSA—specific surface area, ion exchange and retention, surface charge density) and bulk chemical composition where results are invariably confounded to some extent by 129 the presence and properties of impurities. Crystallographic properties of kaolin such as basal spacing, peak shape and crystallinity indices are not very sensitive to the presence of small amounts of impurities. Transmission electron micrographs need to be interpreted with caution as micrometric particles may be impurities rather than being kaolin crystals. This problem can be avoided if individual particles are analyzed by X-ray spectrometry in the TEM (EDS) to confirm that they are kaolin although this is a time consuming process (Singh and Gilkes, 1995). 137

The common pretreatments of soil clay samples consist of 138 (i) digestion in hydrogen peroxide solution at near neutral pH to re- 139 move organic matter although this treatment might oxidize structural 140 ferrous ion in clay minerals (ii) removal of free Fe/Mn oxides by repeat- 141 ed digestion in DCB (dithionite-citrate-bicarbonate) solution which re- 142 duces and dissolves these oxides. The structural Fe in clay minerals can 143 be reduced by this treatment which will then affect the layer charge of 144 the minerals (Stucki, 2006). It is usual to check the purity of separated 145 soil kaolins using XRD but this technique has limited sensitivity 146 especially for highly disordered layer silicates and amorphous 147 alumino-silicates such as allophane. The use of synchrotron XRD can 148 provide greater sensitivity for the recognition of quartz, anatase, rutile, 149 crandallite and other common crystalline impurities in kaolin samples 150 but is not equally effective for determining poorly ordered silicate min- 151 erals. Other analytical techniques such as IR spectrometry and thermal 152 analysis may be used to detect impurities in kaolin concentrates but 153 these techniques are too insensitive to distinguish minor amounts of 154 most mineral species. 155

Thus, it may not be possible to quantitatively determine the amounts 156 of impurity minerals in kaolin concentrates consequently accurate cor- 157 rections of chemical analyses can not be made. The use of Rietveld XRD 158 analysis (Hillier, 2000) in recent years has greatly improved the quanti- 159 fication of minerals in clay concentrates so that reliable correction for 160 some crystalline impurity minerals is now possible; however the sensi- 161 tivity of XRD techniques for minor and disordered constituents remains 162 poor. This is a particular limitation when attempts are made to allocate 163 minor elements (e.g. Cr, Zn, Pb) in kaolin concentrates to mineral spe- 164 cies. Thus, for example it may not be possible to conclude whether µg 165 g^{-1} amounts of Cr are in the structure of kaolin (Singh and Gilkes, 166 1991) or are in minor amounts of associated chromite. Unfortunately, 167 EDS combined with TEM lacks sufficient sensitivity to determine trace 168 levels of metals in single crystals of kaolin. With these reservations in 169 mind, we will now turn our attention to what is known of the properties 170 of soil kaolin. 171

3. Properties of soil kaolin

172

As mentioned in the introduction some mature leached tropical soils 173 may contain large, euhedral kaolin crystals with a high degree of struc- 174 tural order. This kaolin has generally been inherited from deep regolith 175 or sedimentary rocks and does not have a pedogenic origin (Varajão 176 et al., 2001). More commonly kaolin in the solum consists of small crys- 177 tals with a low degree of structural order (Brindley and Brown, 1980). 178 Typical random powder XRD patterns of deferrated kaolin clay from 179 tropical soils are shown in Fig. 2 where only basal (001) and two dimen- 180 sional order (hk) diffraction peaks of kaolin are present. The prism (hkl) 181 reflections that are characteristic of well ordered kaolin are absent due 182 to the high extent of b-axis disorder in soil kaolins. The great width of 183 the basal (001, 002) reflections is predominantly due to the small crys- 184 tal size in the c-axis direction which can be estimated from diffraction 185 line broadening using the Scherrer equation (coherently diffracting do- 186 main size CSD) (Brindley and Brown, 1980). 187

The basal spacing (d (001)) of kaolin appears to increase from 188 0.715 nm to about 0.730 nm as CSD decreases from 40 to 6 nm 189

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Fig. 2. X-ray diffraction patterns of the randomly oriented deferrated clay fraction of red Ultisols, showing the weak broad reflections typical of microcrystalline, b-axis disordered kaolinite with no other clay minerals (K = kaolinite, Q = quartz) (Trakoonyingcharoen et al., 2006).

(Fig. 3). This is an artifact due to displacement of the maximum of the broad 001 reflection that is associated with the steep rise in structure and angular factors with decreasing 20. The data shown in Fig. 3 come from publications on soil kaolin from Indonesia, Thailand, Brazil and Australia together with data for specimen kaolins. Some of the deviation of data from the fitted curve is probably due to differences in the analytical methods used by the different authors.

197The chemical composition of soil kaolin is quite variable and com-198monly does not conform to the ideal composition (39.5% Al₂O₃, 46.6% SiO₂). This diversity is illustrated in Fig. 4 for data for soil kaolin from di-199verse localities. The median value for SiO₂ (46.33%) approximately coin-200 cides with the ideal composition but the median Al₂O₃ concentration is 201 significantly smaller. There is considerable variation about these values 202203which can be ascribed to the presence of impurity minerals and to experimental error but it seems probable that soil kaolin commonly differs 204in composition from the ideal formula. It has been reported that many 205 soil kaolins contain structural Fe with levels of 1-4% Fe₂O₃ being com-206 207monly observed (Hart et al., 2002). Higher values of the iron concentra-208 tion in soil kaolin reported in the literature (up to 11% Fe₂O₃) are probably mostly associated with the presence of iron oxides and other 209 impurities. The iron in kaolin crystals is in octahedral coordination as 210211ferric ions replacing aluminum ions (St. Pierre et al., 1992) a substitution 212 which has no impact on the layer charge but may affect other crystal properties as will be discussed later. 213

Soil kaolin commonly appears to contain potassium at concentrations less than 1% K₂O. It is difficult to determine the location of this potassium but studies by Chi and Eggleton (1999) and Darunsontaya 216 et al. (2012) indicate that minor amounts of muscovite-like mica can 217 occur as interlayers and surface layers on kaolin crystals or as minor 218 amounts of coexisting muscovite that cannot be detected by conven-219 tional or synchrotron XRD techniques. Very detailed studies of soil kao-220 lin using TEM/EDS on ultramicrotomed sections of kaolin crystals have 221 revealed various configurations of muscovite with kaolin. In Fig. 5a-b, 222 the presence of a nanometric crystal of mica (1.0 nm spacing) on the 223 surface of a complex crystal(s) of kaolin (0.7 nm) is revealed. 224

The size and shape of kaolin crystals in tropical soils is highly diverse 225 (Fig. 6a,b,c) with small anhedral platy crystals dominating and with 226 some, tubular and spheroidal crystals. The large euhedral, hexagonal 227 crystals (Fig. 6d) that are characteristic of many industrial and specimen 228 kaolins are uncommon and where they occur in the solum may have 229 been inherited from saprolite or sedimentary rocks. The majority of 230 soil kaolins are mostly composed of small (0.1 µm) platy crystals and 231 these may be anhedral to euhedral hexagonal with all morphologies 232 giving XRD patterns characteristic of a highly b-axis disordered struc-233 ture. There is no relationship between the degree of euhedral character 234 of kaolin crystals and structural order. Some soil kaolins contain a con-235 siderable proportion of tubular (halloysite) or spheroidal crystals and these are commonly associated with soils derived from volcanic detritus 237 or deep regolith (Siradz, 2000; Kew and Gilkes, 2007).

The specific surface area (SSA) of kaolin in tropical soils varies great- 239 ly (10–90 m² g⁻¹) (Fig. 7) and this nearly 10-fold range in SSA is a con- 240 sequence of a similar large variation in crystal size (CSD 001) (Fig. 3). 241 Many industrial and specimen kaolins have much smaller values of 242 SSA. Overall there is no close consistent relationship between SSA and 243 iron content of kaolin but there may be weak positive relationships for 244 individual data sets. The high values of SSA approach those typical of il- 245 lite (40–65 m² g⁻¹) (Dogan et al., 2007) which is a clay mineral that 246 provides substantial cation exchange capacity to some soils (10– 247 40 cmol kg⁻¹) (Borden and Giese, 2001).

There is no systematic relationship between SSA and cation ex- 249 change capacity (CEC) (Fig. 8) as is shown by data for soil kaolin derived 250 from seven publications. These data indicate that the CEC of soil kaolin 251 may be as high as 23 cmol kg⁻¹ which is much higher than values of 252 2–3 cmol kg⁻¹ for standard mineral kaolins (Van Olphen and Fripiat, 253 1979). It is likely that the largest values of CEC shown in Fig. 8 are due 254 to impure samples, and use of inappropriate methodology including 255 measurement of CEC at a high pH where the pH-dependent surface 256 charge of kaolin has increased. The line that describes the lower limit 257 of the data in Fig. 8 corresponds to a surface charge density of 258 0.1 C m⁻² which is smaller than the value of 0.18 C m⁻² determined 259



Fig. 3. Data from published studies on the relationship between the basal spacing (d001) of soil kaolin from tropical soils and crystal size (coherently scattering dimension (CSD 001). Data for standard mineral kaolins are included.

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Fig. 4. The chemical composition of purified soil kaolin. These 176 kaolins were extracted from tropical soils, data from 8 publications and all samples contain minor amounts of impurities (St. Pierre et al., 1992; Melo et al., 2001; Hart et al., 2003; Wiriyakitnateekul, 2005; Kanket, 2006; Trakoonyingcharoen, 2005; Hu and Yang, 2013; Qiu et al., 2014).

by Kanket et al. (2005) for Georgia reference kaolinite although the av-260erage value for these data of about 0.2 C m^{-2} is similar. It seems that the 261 CEC of soil kaolin may mostly reflect the size of crystals rather than the 262263 different natures of the kaolin crystal surfaces contributing to cation exchange, but this interpretation remains speculative. The highly 264disordered internal structure does not appear to be reflected in different 265surface characteristics, possibly because b-axis disorder simply 266translates kaolin layers without modifying them (Brindley and Brown, 2672681980).

Several authors (e.g. Tombácz and Szekeres, 2006) have considered
 the different natures of the two basal surfaces of kaolin crystals (a hy droxyl surface and on oxygen surface) and the complex edge surfaces.
 The relative contributions of the different surfaces to CEC are likely to

the dependent on crystal size and shape. Their different charge properties of these diverse surfaces and their different susceptibilities to 274 charge variation with changing solution pH will have contributed to 275 the very wide range of CEC values reported for soil kaolin which is illustrated in Fig. 8. It is not possible to identify single values of specific surface area (SSA) and cation exchange capacity (CEC) that can be used to 278 represent the kaolin present in highly weathered topsoil in tropical regions. However, SSA values in the range of $20-60 \text{ m}^2 \text{ g}^{-1}$ and CEC values soil kaolins examined by researchers. Values in excess of these numbers might be at least partly due to the presence of impurities and error in measurement. It is clear that industrial and specimen kaolins have SSA and CEC values at the lower limits of these ranges.



Fig. 5. (a) Transmission electron microscope images of a kaolin particle from an oxisol. A basally oriented clay aggregate prepared by the ultramicrotome technique shows a section across kaolin plates; (b) High-resolution electron micrographs of the areas in the white rectangles in (a) showing lattice fringes. Fourier Transform (FFT) images of the areas indicated in (b) show that both 0.7 nm (kaolin) and 1.0 nm (mica) basal spacing minerals are present in this material. Conventional and synchrotron XRDs were unable to detect the 1.0 nm mineral (Darunsontaya et al., 2012).

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Fig. 6. Transmission electron micrographs of soil kaolins (a) WA169, (b) WA235, (c) IN C63 and (d) Georgia reference kaolin G1261 (Hughes et al., 2009). The much larger crystal size of the mineral kaolin is evident.

In addition to the considerable negative charge on the surface of soil kaolin crystals that provides cation exchange capacity there are also positively charged sites that provide anion exchange (AEC) and anion retention (fixation) capacities. As for CEC, the AEC is to some extent pH dependent and also the various contributions of crystal edge and basal surfaces to CEC and AEC remain uncertain (Khawmee et al., 2013). The capacity of soil kaolin to adsorb phosphate is linearly related to the specific surface area (Fig. 9) with kaolins consisting of very small 293 crystals (SSA 80 m² g⁻¹) being able to retain 1000 μ P g⁻¹. For kaolin-294 rich soils, it seems probable that much of the phosphate retention is by 295 kaolin rather than by the more reactive but much less abundant 296 sesquioxides. Adsorbed phosphate may only be sparingly available to 297 plants due to P-fixation by kaolin thereby requiring farmers to apply 298 large amounts of fertilizer. 299



Fig. 7. A plot of specific surface area (SSA) versus iron concentration in kaolin from tropical soils. Data from 13 publications. Data for standard kaolins are included.

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Fig. 8. The non-significant relationship between cation exchange capacity (CEC) and specific surface area (SSA) for kaolin from tropical soils. Data for reference kaolins are included.

300 4. Soil kaolin in relation to soil fertility

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301 If the kaolin in tropical soils had the large crystal size and associ-302 ated low surface reactivity of many mineral kaolins, it is likely that the resultant low chemical fertility of the soil would limit agricultur-303 304 al production and the capacity of the soil to cleanse water. Fortunate-305ly, soil kaolin has more desirable properties and consequently helps 306 support a large proportion of the world's population (Fig. 1). Fertilizers, lime and organic matter are commonly applied to these soils 307 and reactions of these additives with kaolin crystals enhance soil fer-308 tility and structure. 309

An important and intriguing question for the pedologist and soil mineralogist is why do tropical soils usually contain this type of microcrystalline kaolin in the solum? Parent materials may contain large, perfect kaolin crystals but the overlying solum almost invariably contains microcrystalline, structurally disordered kaolin with structural ferric iron. At present, there is no satisfactory explanation for this phenomenon.

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