

## MAGHEMITE FORMATION IN BURNT PLANT LITTER AT EAST TRINITY, NORTH QUEENSLAND, AUSTRALIA

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**Abstract**—Evidence for the formation of maghemite from goethite due to a bushfire on acid sulfate soil at East Trinity, Australia, is presented. Oxidation of pyrite-bearing acid sulfate soils led to precipitation of goethite-impregnated leaf litter. During a major bushfire, goethite with a crystal size calculated from broadening of the 110 reflection of ~9 nm was converted to microcrystalline maghemite (size 12 nm, 220 reflection) and hematite (17 nm, 104 reflection) in a matrix of partly combusted plant litter. Replication of this natural formation of maghemite from goethite was achieved in the laboratory by burning goethite-impregnated leaf litter.

**Key Words**—Acid Sulfate Soils, Bushfire, Charcoal, Goethite, Iron, Maghemite, Organic Carbon.

### INTRODUCTION

Maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) occurs in soils, oxidized igneous rocks and sediments. It is common in soils of subtropical, tropical and humid temperate areas which experience burning in bushfires (e.g. van der Marel, 1951; Bonifas and Legoux, 1957; Schwertmann and Heineman, 1959; Taylor and Schwertmann, 1974; Fitzpatrick, 1978; Coventry *et al.*, 1983; Anand and Gilkes, 1987). Such an occurrence has led to the hypothesis that some maghemite in soils is due to the heating of Fe oxide-rich topsoil. Numerous authors have suggested that the simple heating of Fe oxides (lepidocrocite  $\gamma\text{-FeOOH}$ , or magnetite  $\text{Fe}_3\text{O}_4$ ) produces maghemite, or alternatively, that heating of Fe oxides in organic matter-rich topsoils (goethite  $\alpha\text{-FeOOH}$ , or ferrihydrite  $5\text{Fe}_2\text{O}_3 \cdot 9\text{H}_2\text{O}$ ) provides some reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  which leads to the formation of maghemite rather than hematite (e.g. Fitzpatrick, 1985; Schwertmann, 1985; Schwertmann and Cornell, 2000; Cornell and Schwertmann, 1996). Other means of maghemite formation are the aerial oxidation of magnetite in soils derived from mafic rocks (Fontes and Weed, 1991) and of magnetite of microbial origin. In general, the formation of maghemite represents a solid-state process involving internal rearrangements within a solid precursor Fe mineral, and this formation may be facilitated by the presence of  $\text{Fe}^{2+}$  ions which occupy specific structural sites in the inverse spinel structure (Cornell and Schwertmann, 1996).

The postulated formation processes are supported by laboratory synthesis of maghemite through the heating of Fe oxide minerals. Maghemite formation has been induced by heating of lepidocrocite or magnetite at

250°C. Maghemite may also be formed by heating goethite or ferrihydrite at >300°C in the presence of organic material (Cornell and Schwertmann, 1996; Campbell *et al.*, 1997). These heating experiments have also shown that in the presence of organic matter goethite and ferrihydrite alter to maghemite rather than to hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ). However, there have been few documented observations of the formation of maghemite by fire under natural conditions (e.g. Longworth *et al.*, 1979; Stanjek, 1987; Kletetschka and Banerjee, 1995; Morinaga *et al.*, 1999; Houyuan *et al.*, 2000), and such observations are usually circumstantial rather than direct. For instance, Ketterings *et al.* (2000) reported that slash-and-burn fires in Indonesia resulted in the burning of topsoil and appeared to convert goethite to ultra-fine maghemite.

The East Trinity site is a drained forested wetland adjacent to the city of Cairns in tropical Far North Queensland, Australia. Oxidation of sulfide minerals has led to the development of extensive acid sulfate soils and deposition of secondary Fe minerals (goethite, ferrihydrite, jarosite) in soils and flooded areas. A major bushfire engulfed part of the East Trinity site and resulted in mineralogical transformations in the topsoil. Laboratory and field investigations were conducted on the litter layer and the topsoil prior to and after the bushfire. Results of this work provide conclusive evidence for maghemite formation in East Trinity soils by thermal alteration of precursor goethite.

### MATERIALS AND METHODS

#### *Sampling and sample preparation*

Leaf litter and topsoil samples (top 2 cm) were collected before and after the fire. Three Fe oxide-rich leaf litter samples were taken as spot samples, and small amounts of Fe oxide precipitate were collected at the base of plastic piezometers which were installed to

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monitor groundwater (Hicks *et al.*, 1999). All samples consisted of a mixture of mineral and plant material but less organic material was present in partly combusted leaf litter.

#### *Mineralogical and chemical analyses*

Iron oxide-rich materials were handpicked from the litter for mineralogical, microscopic and chemical analyses. The mineral constituents were identified by X-ray powder diffraction using a Siemens D5005 diffractometer with CuK $\alpha$  radiation and a crystal monochromator at the Advanced Analytical Centre, James Cook University (AAC, JCU). Because of the small amounts of material available for some samples, a low-background, non-diffracting single crystal was employed as a substrate for powders to reduce background noise. However, a fracture and associated displacement in the crystal caused enhanced scattering in the <2.1 Å range preventing interpretation of patterns at high  $2\theta$  angles. Samples were ground by hand and distilled water was added via a pipette. A slurry of mineral and plant materials sufficient to give a very thin layer of dry material was pipetted onto the crystal substrate and analyzed from  $7^\circ$  to  $40^\circ 2\theta$  at a scan rate of  $0.25^\circ \text{ min}^{-1}$ . The crystal size (*i.e.* coherently diffracting domain size) of Fe oxides was calculated from diffraction line broadening of major reflections using quartz reflections to indicate instrumental broadening (Klug and Alexander, 1974).

The morphology of individual Fe oxide crystals and aggregates was studied using transmission electron microscopy (TEM) at the University of Newcastle. One drop of suspension was placed on a carbon film supported by a 200 mesh copper grid. The samples were examined in a JEOL 1200-EX transmission electron microscope using an acceleration voltage of 80 kV. Scanning electron microscopy (SEM) was conducted on carbon- and gold-coated samples using JEOL JSM-6300 and JEOL JSM-5410LV scanning electron microscopes at the AAC, JCU. Qualitative energy dispersive spectrometry (EDS) analyses were performed on selected goethite and maghemite-hematite rich materials using a JEOL JSM-6300 scanning electron microscope with an attached X-Flash energy dispersive detector at the AAC, JCU.

Chemical analyses of materials followed Australian standard procedures (Raymond and Higginson, 1992). Bulk original and burnt samples were dissolved in HF-HNO<sub>3</sub>-HClO<sub>4</sub> and analyzed by inductively coupled plasma atomic emission spectrometry (ICPAES) for their Al, As, Cu, Fe, Mn, P, Pb and Zn contents. Organic carbon was determined using a Leco furnace.

#### *Heating experiments*

Leaf litter samples were homogenized briefly in a chrome-steel ring mill, and then split into subsamples for four heating experiments. Each experiment used separate

samples of ~40 g each. Samples were placed in a porcelain crucible and covered with a porcelain lid to prevent spattering. In the first experiment, the porcelain containers were placed in a muffle furnace, and the temperature was then raised from ambient temperature to 300°C at a rate of  $10^\circ \text{ min}^{-1}$ . In the second experiment, the porcelain crucibles were placed in the furnace once it had been heated to 300°C. In both experiments, the samples remained in the furnace for 4 h at 300°C. In the third experiment, the porcelain crucibles were placed in the muffle furnace, and the temperature was then raised from ambient temperature to 400°C at a rate of  $10^\circ \text{ min}^{-1}$ . In the fourth experiment, the porcelain crucibles were placed into the furnace once it had been heated to 400°C. In both of the last two experiments, the samples remained in the furnace for 1 h at 400°C. The crucibles were removed from the furnace to cool with their lids on. Some sample material from the heating experiments was used for TEM and SEM studies without further sample processing. Remaining sample portions were briefly ground in a chrome-steel ring mill for XRD and chemical analyses as described previously.

## RESULTS AND DISCUSSION

#### *Preburning soils*

Abundant Fe oxide-rich leaf litter was noted in low-lying areas of the southwestern East Trinity site. The material consisted of yellowish mustard and orange-yellow coloured (Munsell colour 3/4 2.5YR) coatings and impregnations on grasses, tree roots, leaves, bark and fallen branches (Figure 1a). These colours are consistent with the presence of goethite with lesser ferrihydrite (Fitzpatrick 1985). The Fe oxide-rich leaf litter formed a blanket of ~2 cm thick. Iron oxide impregnations and encrustations were particularly abundant in the uppermost portion of the litter and were also observed to a limited degree in the top 2 cm of the underlying acid sulfate soil. Encrustations were more abundant in those areas that were noticeably drier than the surrounding areas (*i.e.* around the bases of trees as opposed to intermittent creeks that drain the area). The use of a pocket magnet indicated the absence of magnetic minerals within the Fe oxide-rich leaf litter.

The presence of Fe oxide as a surficial encrustation is related to seasonal flooding and seawater incursions into low-lying areas because more elevated soils did not exhibit the Fe oxide encrustations. The Fe oxide is likely to have formed from Fe released by oxidation of acid sulfate soils on the site which generate ferrous-rich, acidic soil waters at depth. These acid solutions seep from higher areas into creeks, drains and surface pools. Also, the acid waters were brought to the surface as a result of rising water tables during heavy rainfall and/or seawater incursions. Precipitation of Fe oxides occurred at seepage points and in surface waters due to the oxidation of ferrous to ferric ions and the neutralization

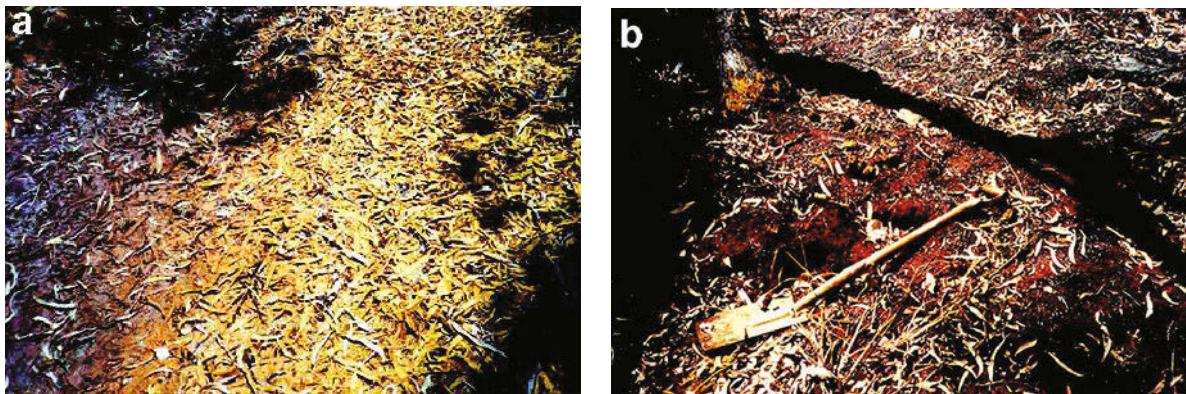


Figure 1. Field photographs: (a) blanket of leaf litter impregnated with goethite and possibly ferrihydrite; (b) ash blanket with abundant maghemite-hematite (field of view ~4 m wide).

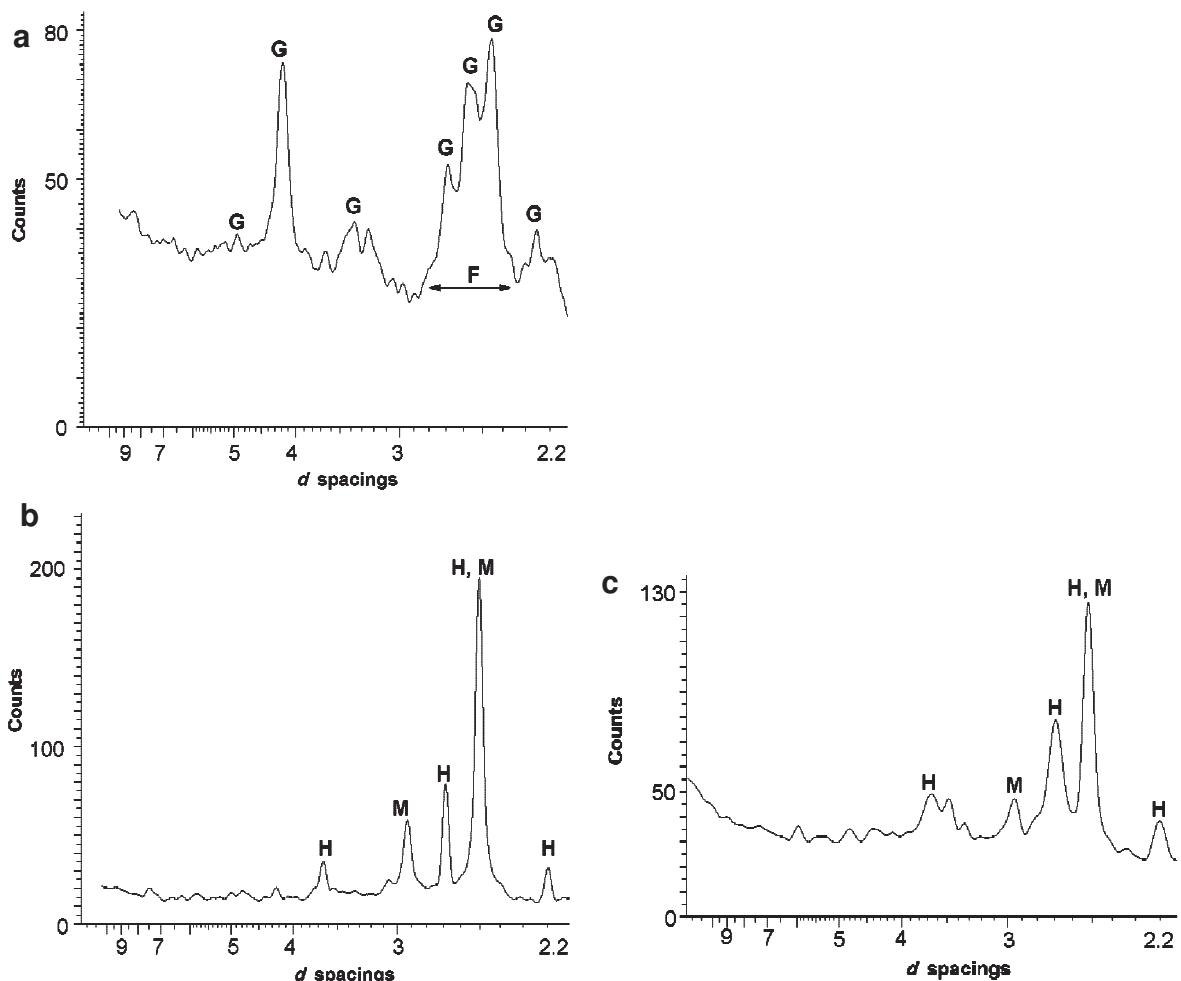


Figure 2. XRD patterns of samples on a low-background holder. Reflections due to goethite, hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ), and maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) are indicated together with the location of the broad ferrihydrite reflection that may be concealed by broad goethite reflections. Other reflections are due to soil minerals given in the text. (a) Iron oxide precipitate collected from the surface of a plastic pipe in the litter layer; (b) ash layer after partial combustion of litter in the field; and (c) ash from partial combustion of litter in the laboratory. F: ferrihydrite; G: goethite; H: hematite; M: maghemite.

of acid drainages with neutral-alkaline surface waters. During the retreat of floodwaters, the surface of topsoils, creek beds and leaf litter becomes heavily encrusted with Fe oxide.

The diffraction pattern of Fe oxide precipitate from the surface of the plastic pipe was more intense and indicated that the material consisted mostly of goethite

and possibly some ferrihydrite (Figure 2a) although no discrete reflections of this mineral were resolved. The XRD patterns of many subsamples of Fe oxide-rich leaf litter samples are of low intensity due to the presence of leaf material but do show major amounts of goethite and minor and various amounts of quartz, kaolinite, plagioclase, illite and halite. Spacings for goethite correspond

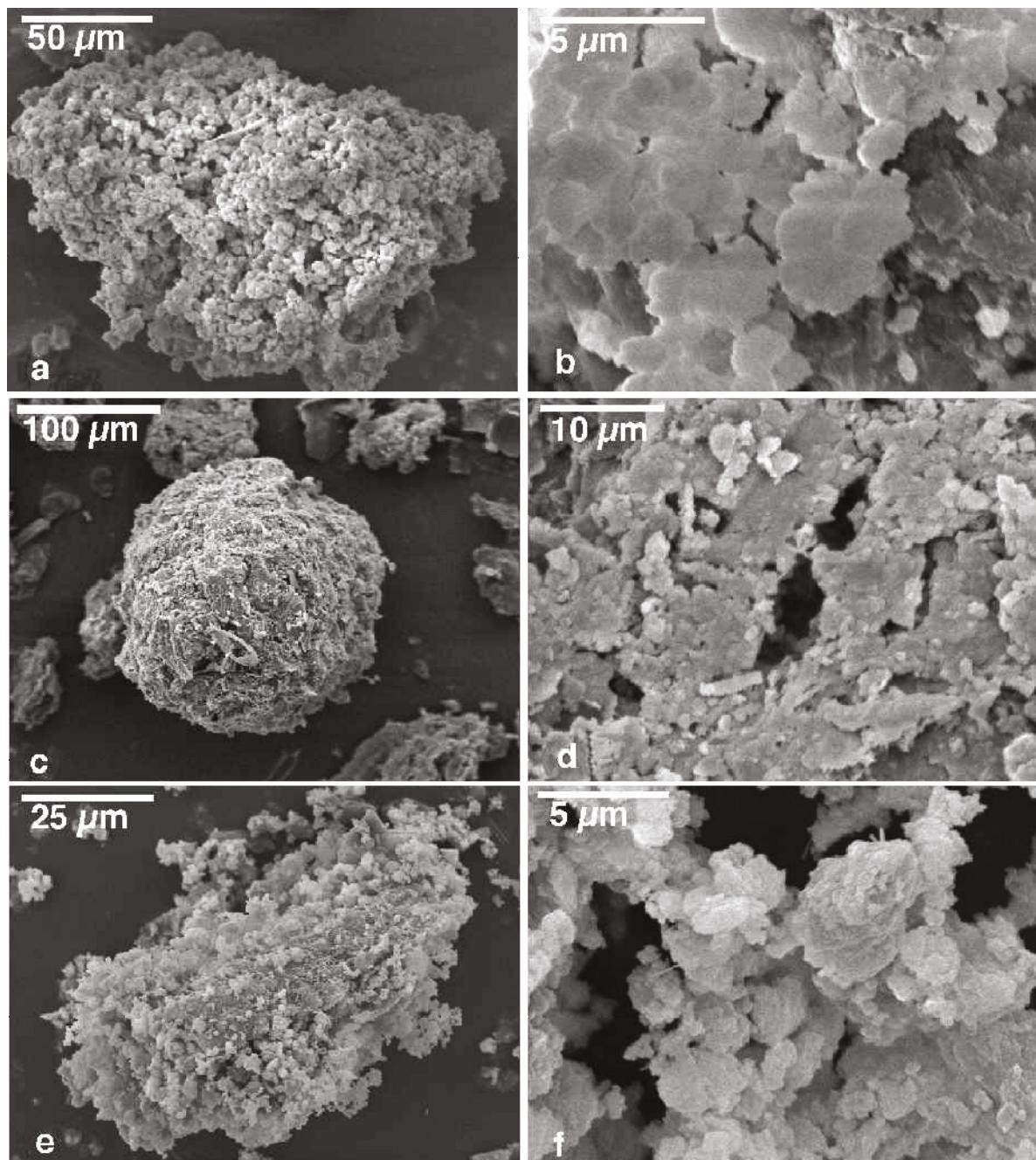


Figure 3. SEM images: (a, b) angular goethite (and possible ferrihydrite) aggregate displaying platy Fe oxide grains; (c,d) subrounded maghemite-hematite aggregate collected after the bushfire displaying angular, platy and rod-like Fe oxide grains; (e,f) irregularly shaped maghemite-hematite aggregate from the heating experiments displaying stacked, platy Fe oxide grains.

to ideal goethite with no Al substitution, and the crystal size (coherently diffracting zone, 110 reflection) of the goethite is small (9 nm). The complex aggregates seen in electron photomicrographs consist of clusters of these submicron-sized crystals. Some Fe oxide aggregates reach diameters of 10 µm and are themselves loosely packed together to form subrounded aggregates up to 260 µm in diameter (Figures 3a,b, 4a). In addition, broken and complete siliceous diatom plates were present in the larger aggregates. The EDS analyses of aggregates revealed the presence of major amounts (*i.e.* >100 g kg<sup>-1</sup>) of Al, Si and S, and minor amounts (*i.e.* 1–100 g kg<sup>-1</sup>) of K, Ca, Mg, P and Cl reflecting the microscopic admixture of Fe oxides, quartz, kaolinite, plagioclase, illite and halite.

Chemical analyses of bulk Fe oxide-rich leaf litter (Table 1) showed that these materials contain major amounts (*i.e.* >100 g kg<sup>-1</sup>) of Fe and C<sub>org</sub>, minor amounts (*i.e.* 1–100 g kg<sup>-1</sup>) of Al, Ca, Cl, K, Mg, Na, S and Si, and traces (*i.e.* <1 g kg<sup>-1</sup>) of As, Cu, Mn, P, Pb and Zn (Table 1). Some of these elements may be adsorbed onto or incorporated into the Fe oxides which have a large, reactive surface area capable of adsorbing both cations and anions (Cornell and Schwertmann, 1996). These elements are also present in the various soil minerals identified above and in the leaf litter as some of them (Ca, Cu, Fe, K, Mg, Mn, P, S, Si, Zn) are essential plant nutrients.

#### Burnt soils

Burning of the southeastern section of the East Trinity site resulted in much of the leaf litter deposited on the site being combusted. In its place was a rusty red-brown colored material (Munsell colors 6/6 10YR, 4/6 5YR) consisting mostly of a charred litter, charcoal and ash (Figure 1b). This blanket had a depth of ~2 cm and contained a magnetic component. The red-brown colored magnetic material was also dominant within the top 2 cm of the soil with minor occurrences to 5 cm depth.

The red-brown material in the ash layer contained much hematite and maghemite with minor amounts of quartz and clay minerals (Figure 2b). The XRD peaks of both Fe oxides are broad indicating that the crystal size of the hematite (104 reflection) is ~17 nm and maghemite (220 reflection) is ~12 nm. These sizes are larger than for the precursor goethite (9 nm for the 110 reflection) which may indicate that the goethite particles consisted of two or more discrete coherently diffracting zones. Some of the Fe<sub>2</sub>O<sub>3</sub> minerals may have formed

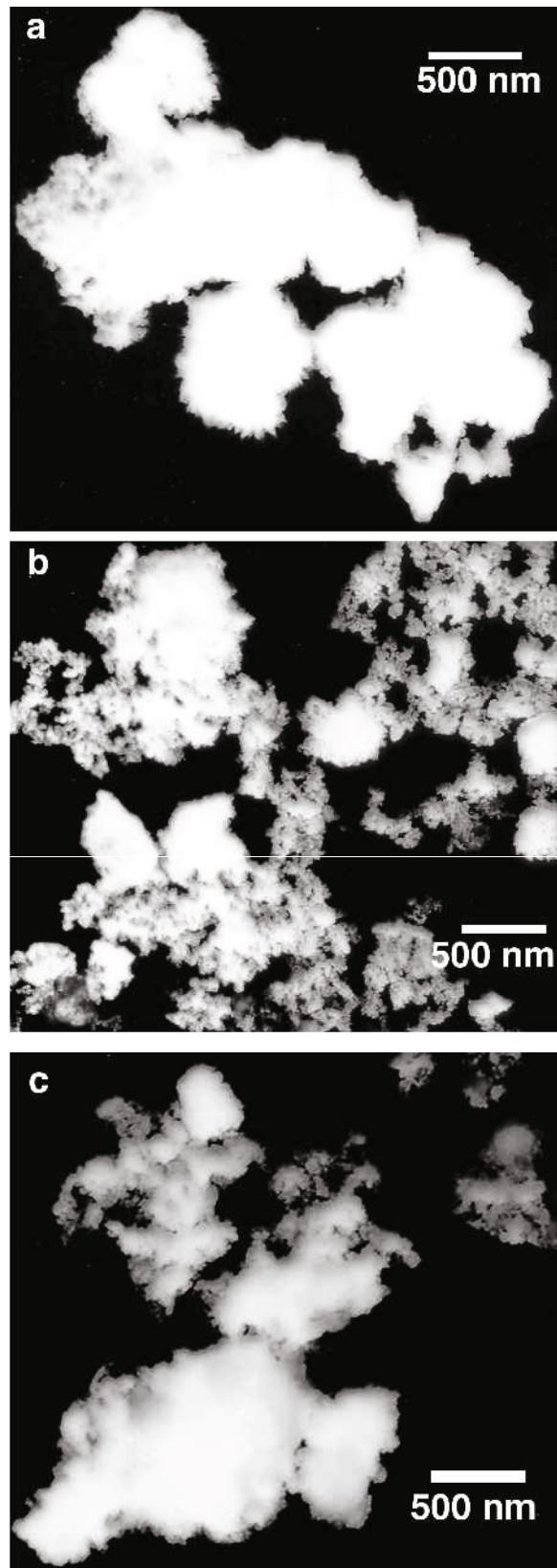


Figure 4. TEM images: (a) porous goethite (and possible ferrihydrite) aggregate displaying a rim of acicular needles; (b) porous maghemite-hematite aggregate collected after the bushfire displaying larger Fe oxide grains enclosed by smaller Fe oxide phases; (c) maghemite-hematite sample from the heating experiment showing Fe oxide crystals with an outer rim of fine-grained Fe oxides.

Table 1. Chemical analyses of goethite-rich leaf litter, maghemite-hematite-rich ash layer, and synthesized maghemite-hematite-rich ash.

	Al (g kg <sup>-1</sup> )	As (mg kg <sup>-1</sup> )	Cu (mg kg <sup>-1</sup> )	Fe <sub>total</sub> (g kg <sup>-1</sup> )	Mn (mg kg <sup>-1</sup> )	P (mg kg <sup>-1</sup> )	Pb (mg kg <sup>-1</sup> )	Zn (mg kg <sup>-1</sup> )	C <sub>org</sub> (g kg <sup>-1</sup> )
Goethite-rich leaf litter; mean ( <i>n</i> = 4)	6.9	55	7	209	79	429	20	18	150
Maghemite-hematite-rich ash layer; mean ( <i>n</i> = 4)	33.4	61	16	261	82	1002	16	22	34.9
Synthesized maghemite-hematite-rich ash; mean ( <i>n</i> = 6)	19.7	82	13	312	92	619	12	32	78.9

from ferrihydrite and consist of smaller crystals than the Fe<sub>2</sub>O<sub>3</sub> formed from goethite. The spacings of both hematite and maghemite corresponded to ideal  $\alpha$ - and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> indicating that little or no substitution of Al or Fe<sup>2+</sup> occurred (Brindley and Brown, 1985). The XRD pattern of maghemite did not exhibit the superstructure lines listed by Brindley and Brown (1985). The Fe oxides occur as subrounded to spherical aggregates up to 140  $\mu\text{m}$  in diameter (Figures 3c,d, 4b). Charcoal fragments and siliceous diatoms are embedded in the aggregates. In addition to Fe, the aggregates contain Si and Al with traces of S, K, P, Cl and Ca.

Bulk chemical analyses show that the maghemite-hematite materials contain less C<sub>org</sub> and more metallic elements than the original leaf litter (Table 1) due to the loss of some carbon and water during the bushfire.

#### Heating experiments

Heating experiments were conducted on three different goethite-rich leaf litter samples in order to demonstrate that combustion of goethite-rich leaf litter results in the formation of a maghemite-hematite mixture. None of these samples contained magnetic minerals.

The various heating experiments yielded ash of very similar mineralogy, morphology and chemistry to the naturally burnt materials. For each heating regime, the synthesized products consisted mostly of maghemite and hematite (Figure 2c). The XRD peaks for the Fe oxides are broader than those for the natural ash with crystal sizes (coherently diffracting domain size) of ~6 nm for hematite (104 reflection) and 9 nm for maghemite (220 reflection). These sizes are similar to the value of 9 nm for the 110 reflection of precursor goethite. Both minerals had spacings corresponding to pure Fe<sub>2</sub>O<sub>3</sub>, and there were no superlattice reflections for maghemite. The Fe oxides are present as irregularly shaped, angular and subrounded aggregates up to 260  $\mu\text{m}$  in diameter (Figures 3e,f, 4c). Pore space is highly variable and grain aggregates range from solid to loosely packed. The EDS analyses revealed the presence of Al, Ca, Cl, K, Mg, Na, P, S, Si and Ti in the Fe oxide-rich aggregates.

The synthesized samples have a chemical composition similar to the natural maghemite-hematite materials (Table 1), although the C<sub>org</sub> concentration of synthesized samples (mean 78.9 g kg<sup>-1</sup>) is distinctly higher than for the field samples of maghemite-hematite ash (mean 34.9 g kg<sup>-1</sup>). This difference is presumably due to restricted combustion of organic matter in the closed porcelain crucibles.

#### Maghemite formation

This paper provides clear evidence for the formation of maghemite in soils by partial combustion of goethite-impregnated plant litter. The favorable circumstance of the litter layer being a discrete material on the soil surface means that maghemite has not formed from oxidation of magnetite in the underlying soil, a circumstance that confounds some other reports of maghemite formation by heating of soils. The partial combustion of the organic litter layer in both the naturally and laboratory combusted materials may indicate that reducing conditions prevailed in some portion of the material during combustion. Under these circumstances dehydroxylation of goethite may have been associated with generation of some Fe<sup>2+</sup> that could favor development of the inverse cubic spinel structure  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> with minor solid-solution with Fe<sub>3</sub>O<sub>4</sub> (magnetite) rather than development of the hexagonal  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> structure. Spacings of XRD reflections were identical to those for ideal  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> (Brindley and Brown, 1985) so that any solid substitution by Fe<sub>3</sub>O<sub>4</sub> would have been minor (<10%). Ferrous iron could not be determined chemically due to the presence of organic carbon in the samples.

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