

Synthetic Iron Oxides as an Indicator of Reduction in Soils (IRIS)

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ABSTRACT

Various circumstances in wetland delineation or mitigation strategies require the documentation of reducing conditions in soils. An innovative approach for assessing reduction in soils known as Indicator of Reduction in Soils (IRIS) has been recently introduced. IRIS tubes are manufactured by coating polyvinylchloride (PVC) pipe with a paint prepared from an Fe oxide (mainly ferrihydrite) suspension. When the iron oxide is synthesized by adding KOH to a FeCl₃ solution, the mineral phase is essentially pure ferrihydrite. Over time, and under particular conditions, the ferrihydrite is transformed to more crystalline forms such as goethite and hematite. The objectives of this study were (i) to assess the mineralogical composition of iron oxide paint synthesized for the manufacture of IRIS tubes and (ii) to assess the significance of mineralogical composition of the Fe oxide paint on desirable qualities for the construction of IRIS tubes. The effects of pH and storage time on the mineralogical alteration of ferrihydrite are examined and compared with performance of the paint by a scale for adhesion and durability. Paint composed of nearly pure ferrihydrite shows poor adhesion and durability. Data suggest that for paint to adhere well to the PVC tubing, the Fe oxide suspension must contain a minimum of 30 to 40% goethite. This desired paint composition can be facilitated during synthesis by titrating the Fe oxide suspension to pH 11 or 12 rather than pH 7.5, which is typically prescribed for the formation of synthetic ferrihydrite.

DOCUMENTATION of reduced soil conditions is needed or desired for a number of purposes. For example, wetland construction or restoration projects associated with mitigation efforts usually have hydrological performance standards, but there is an increasing demand for standards linked to wetland function, such as demonstrating that soil conditions are reducing. There are also numerous applications linked to hydric soil assessment and the Technical Standard (TS) for Hydric Soils. The TS requires a demonstration both that the soil is saturated and reduced (National Technical Committee for Hydric Soils, 2000). In the evaluation or testing of Field Indicators of Hydric Soils (USDA-NRCS, 2005), the TS must be met. In cases where a soil does not meet a field indicator, the TS can be used to demonstrate that a soil is hydric. In these situations, the soil must be shown to be reducing.

The generally accepted approach to demonstrate reducing conditions in soils is either (i) to measure the redox potential with Pt electrodes and to show that data plot in the "reducing" zone in an Eh-pH diagram or (ii) to apply α , α dipyridyl to the soil and observe a positive test (pink color) for ferrous iron. Both of these ap-

proaches have limitations because of either the need for specialized equipment or the difficulty in obtaining the necessary chemicals. These limitations have led to recent interest in IRIS tubes as an alternative approach for confirming reducing conditions in soils. The idea was conceived by Byron Jenkinson, whose experimentation and testing of IRIS tubes formulated the basis for his PhD dissertation (Jenkinson, 2002). Others have begun to evaluate IRIS tubes to try to correlate their performance to other accepted standards of demonstrating reduction in soils (Castenson, 2004; Jenkinson and Franzmeier, 2006; Castenson and Rabenhorst, 2006; Rabenhorst and Castenson, 2005).

In Jenkinson's original dissertation work, he gave considerable attention to the construction of the tubes themselves as well as some discussion of the nature of the paint used to prepare the tubes (2002.) Relatively little attention, however, has been given to the mineralogical nature of the paint. Incidental observations during the course of our studies led us to question the importance of the mineralogical nature of the paint, both in regard to its function and to its properties, which affect fabrication or construction of the IRIS devices. Therefore, the objectives of this study were (i) to assess the mineralogical composition of iron oxide paint synthesized for the manufacture of IRIS tubes for documenting reduced soil conditions and (ii) to assess the significance of mineralogical composition of the Fe oxide paint on desirable qualities for the construction of IRIS tubes.

MATERIALS AND METHODS

IRIS Tube Construction and Ferrihydrite Paint Synthesis

IRIS tubes are prepared by applying an iron oxide paint to lengths of PVC pipe that are approximately 21 mm in diameter (typically 0.5-inch schedule 40 PVC pipe). Preparation of the paint has been described by Jenkinson (2002), wherein the iron oxide (mainly ferrihydrite) is precipitated from a 0.5 M ferric chloride solution by adding 1 M KOH until a pH of 7 to 8 is reached. This is a slight modification from the procedure of Schwertmann and Cornell (2000) for the synthesis of 2-line ferrihydrite, wherein they prescribe addition of 330 mL of 1 M KOH to 0.5 L of 0.2 M ferric nitrate to reach the same pH of between 7 and 8. Excess salts are then removed by centrifugation and dialysis, and the suspension is evaporated to the appropriate consistency for applying to the tubes. The paint is stored in the dark to minimize mineralogical alteration over time.

Previous IRIS tube experiments were conducted during the spring of 2003 (Castenson, 2004; Castenson and Rabenhorst, 2006), in late winter and spring of 2004 (Rabenhorst and Castenson, 2005) and also during the summer and fall of 2004. Therefore, iron oxide paint was available in storage that had been synthesized in January 2003, January and May 2004, as

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Published in Soil Sci. Soc. Am. J. 70:1227-1236 (2006).

Wetland Soils

doi:10.2136/sssaj2005.0354

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Abbreviations: IRIS, Indicator of Reduction in Soils; PVC, polyvinylchloride; TS, Technical Standard.

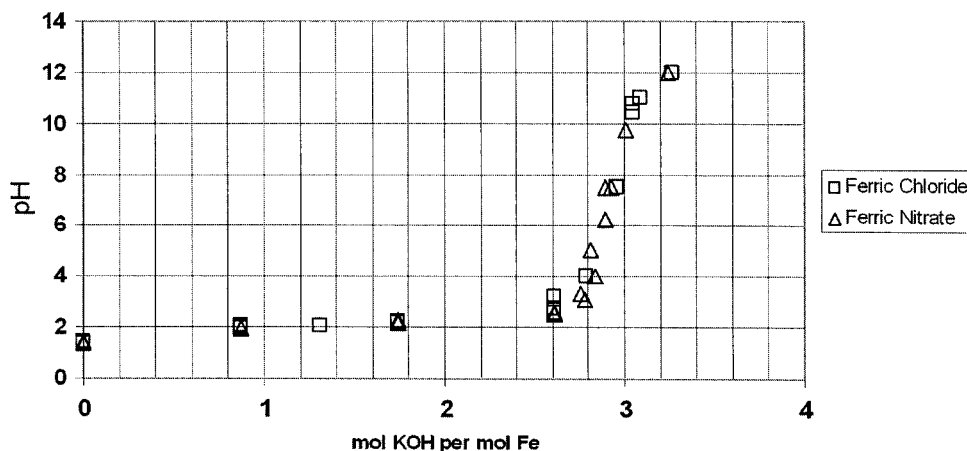


Fig. 1. Titration curve of pH versus quantity of base added per mol of ferric salt. Note that both chloride and nitrate salts behave identically.

well as paint freshly made in mid September 2004. In actuality, the pH of the Fe oxide paint suspension was not monitored carefully during synthesis, and it is likely that the pH may have been higher than 8. Four paint samples were examined that had been in storage for periods of 14, 120, 270, and 625 d. These materials were freeze dried, crushed with an agate mortar and pestle, and then stored dry until analyzed.

The colors of dry samples were measured with a Minolta CR-300 digital colorimeter. Total iron oxides (Fe_a) were extracted from triplicate 0.05-g samples of the paint following a modification of the dithionite–citrate–buffer procedure of Fanning et al. (1970) adjusted to accommodate smaller sample sizes. Poorly crystalline iron oxides (Fe_o) were measured following a procedure similar to 6C6 of the National Soil Survey Laboratory (Soil Survey Staff, 1996). Triplicate 0.1-g samples were extracted with 200 mL of 0.2 M ammonium oxalate ($(NH_4)_2C_2O_4$ adjusted to pH 3 with 0.2 M oxalic acid ($H_2C_2O_4$) in the dark for 4 h. Iron in the extracts was measured by atomic absorption spectroscopy.

X-ray diffraction scans were collected on randomly oriented powder mounts of the paint samples with a Philips PW1830 X-ray diffractometer equipped with a Cu tube and a curved crystal graphite monochromator. Samples were scanned from 10 to $80^\circ 2\theta$ at a rate of $0.75^\circ 2\theta \text{ min}^{-1}$.

Experimentation on pH and Storage Time Effects

When constructing IRIS tubes during the summer of 2004 and the spring of 2005, we repeatedly experienced problems with newly synthesized paint that had a dull luster and did not

adhere well to the PVC tubing, even after thorough cleaning and sanding as described by Jenkinson (2002). Eventually, we realized that using newly synthesized paint always resulted in a dull luster and poor adhesion. However, if we waited a few weeks before using the paint, it sometimes had a more shiny luster and adhered better to the tubing. We postulated that this improvement might be the result of mineralogical alterations that were occurring in the Fe oxide suspensions over time as they were being stored. We therefore designed an experiment to evaluate this hypothesis.

Suspensions of ferrihydrite were prepared following the specifications of Schwertmann and Cornell (2000) and Jenkinson (2002) by titrating 0.5 M solutions of $FeCl_3$ or $Fe(NO_3)_3$ to pH 7.5 with 1 M KOH. After centrifugation and dialysis, suspensions were stored in the dark. Approximately 30 to 40 mL of the suspensions were removed and immediately freeze dried after periods of 4, 7, 14, 21, 28, 42, and 56 d. Because it is reported that the storage pH of a ferrihydrite suspension affects the degree to which it is transformed to more thermodynamically stable species (Schwertmann and Cornell, 2000; Cornell and Schwertmann, 2003), suspensions of ferrihydrite were also synthesized by titrating 0.5 M solutions of $FeCl_3$ or $Fe(NO_3)_3$ to pH values of 4.0, 11.0 and 12.0 with 1 M KOH. These suspensions were also treated, stored, and sampled as described above. The pH titration curves for both the chloride and nitrate salts were nearly identical (Fig. 1). The ferrihydrite typically began to precipitate around pH 4, where the titration curve becomes very steep. One can also see from Fig. 1 that the target pH values of 4.0, 7.5, 11.0, and 12.0 are all along the steep portion of the titration curve. Thus, the quantity of base added to move

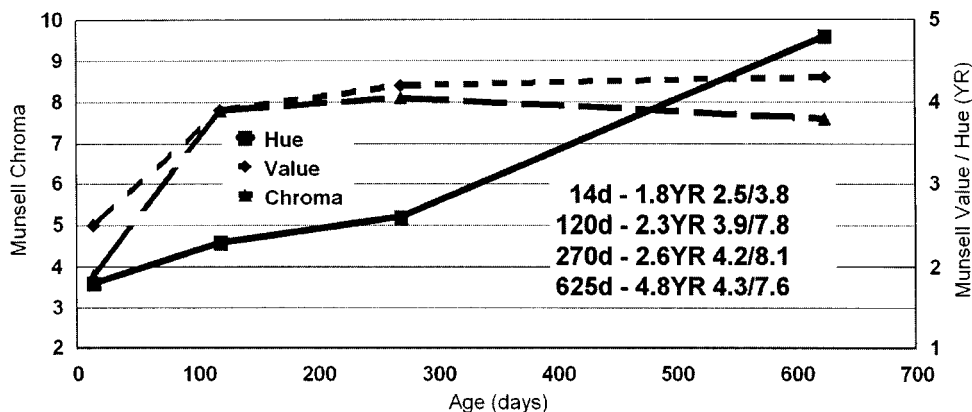


Fig. 2. Munsell dry color of synthetic Fe oxides stored as aqueous suspensions for various periods of time.

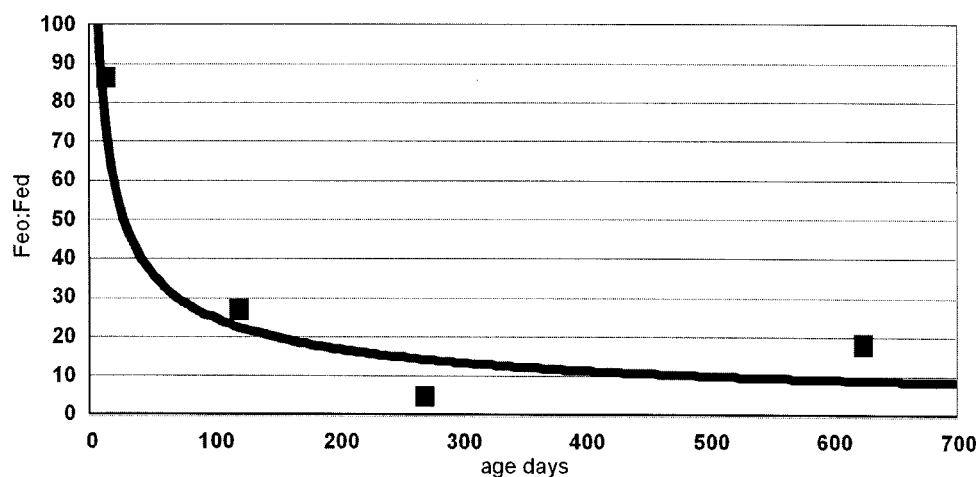


Fig. 3. Ratio of oxalate extractable Fe (Fe_o) to dithionite extractable Fe (Fe_d) (as percent) for synthetic Fe oxides stored for various periods of time (in the dark) as aqueous suspensions. pH was not monitored carefully during the synthesis of the three older iron oxide samples.

between pH 4.0, 7.5, 11.0, and 12.0 was approximately 0.12, 0.15, and 0.25 mol per mol Fe, which represents a relatively small portion of the approximately 3.5 mol of base added per mol Fe. Total Fe was extracted from duplicate 50-mg samples with 6 M HCl, and acid ammonium oxalate extractions were made in the dark with duplicate 50-mg samples and 100 mL of $(NH_4)_2C_2O_4$ adjusted to pH 3. Mineralogy of the samples was evaluated by X-ray diffraction.

Following periods of 4, 7, 14, 21, 42, and 56 d, each of the synthesized paint samples was applied to a section of PVC tubing with a soft foam brush while the PVC tube was being rotated on a lathe-like device (in the manner normally used in constructing IRIS tubes). After drying for 24 h, the durability and adhesion of the paint to the PVC tubing was evaluated by wiping one's index finger a single time along a portion of the painted and dried tube and by the following numerical scale from 1 to 5: 1—paint wipes off when applying very slight pressure; 2—paint wipes off when applying slight pressure; 3—paint wipes off when applying moderate pressure; 4—paint wipes off only when applying firm pressure; 5—paint does not wipe off when applying firm pressure.

RESULTS AND DISCUSSION

Initial Examination of Paint of Various Ages

Dry colors measured on the four initial paint samples of different ages are shown in Fig. 2. As the paint aged from 14 to 120 d, dramatic increases in Munsell chroma (4 units) and value (3 units) were observed, resulting in a paint color that becomes both brighter and lighter. Changes in these parameters between Day 120 and 625 were relatively small (<1 unit). In contrast, Munsell hue changed only half a unit over the first 120 d but continued to change a total of 3 units over the period of 625 d, gradually becoming more yellow. Because iron oxide colors have been shown to reflect mineralogy (Schwertmann, 1993; Bigham et al., 2002), these changes in colors of the paint were thought to reflect possible changes in mineralogy.

Dithionite extractable iron (Fe_d) is generally understood to represent total extractable iron oxides, and acid ammonium oxalate extractable iron (Fe_o) represents poorly crystalline iron oxides (Schwertmann and Taylor, 1989) such as ferrihydrite. Therefore, the ratio of Fe_o

to Fe_d can give some indication of the mineralogical nature of the iron oxides. Figure 3 shows the ratio of Fe_o to Fe_d for the four iron oxide paint samples. Not surprisingly, the youngest paint (14 d) is predominantly (88%) Fe_o , which suggests that it retains much of the character of the originally synthesized ferrihydrite. Cornell and Schwertmann (2003) report that, across a wide range of temperatures and pH, the transformation rate of ferrihydrite to the more crystalline phases follows first order kinetics, indicating that the rate is determined by the quantity of ferrihydrite remaining. The $Fe_o:Fe_d$ ratio in the samples decreased rapidly over time such that paint stored for 120 d or longer was mostly not oxalate extractable ($Fe_o:Fe_d$ of 6–30%). The rate of transformation also increases at warmer temperatures (Cornell and Schwertmann, 2003), so that storage under refrigerated conditions would be expected to slow the alteration. Some of the variation exhibited in Fig. 3 (and in Table 1) is likely the result of the paint samples being synthesized by different workers and without careful pH controls during the titration with KOH.

To identify the particular mineral phases present, samples were scanned by X-ray diffraction (Fig. 4). The youngest material is poorly crystalline and mainly shows two very broad and weak peaks at 0.26 and 0.15 nm, typical for 2-line ferrihydrite, although hints of goethite peaks at 0.42, 0.33, and 0.23 nm can also be discerned. This corresponds well to an $Fe_o:Fe_d$ ratio of 88%, which suggested that the material was dominantly ferrihydrite. In the other three samples that are 120 d or older, strong peaks for both hematite and goethite are plain while the broad ferrihydrite peaks are obscured. On the basis of

Table 1. Proportions of mineral phases in four samples of iron oxide paint ranging in age from 14 to 625 d.

Age	Phase		
	Ferrihydrite	Goethite	Hematite
d	%		
14	88	12	0
120	30	37	33
270	6	59	35
625	20	42	38

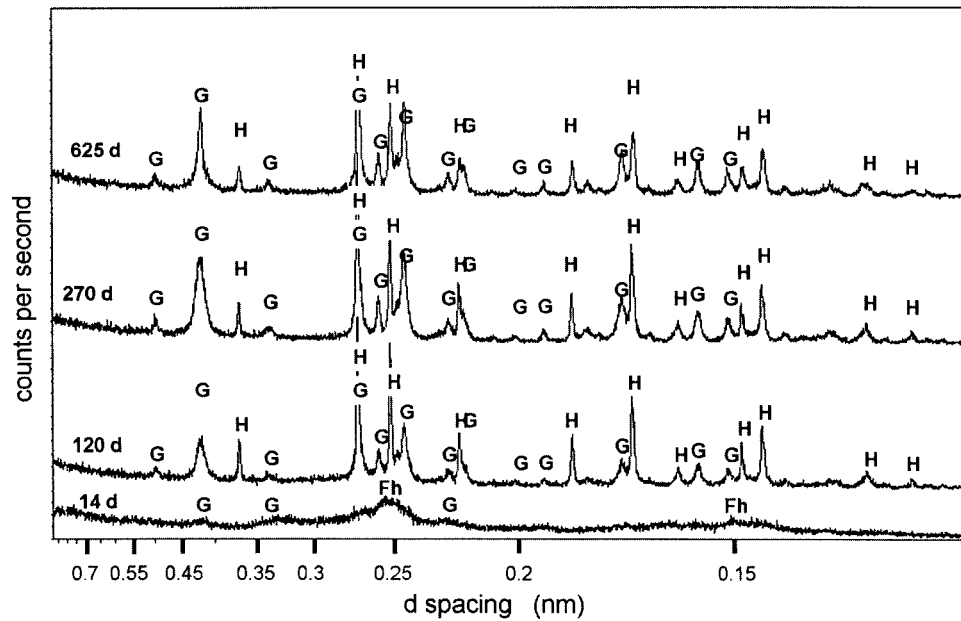


Fig. 4. X-ray diffractograms of synthetic Fe oxides stored as aqueous suspensions (in the dark) for various periods of time. Fh—ferrihydrate; G—goethite; H—hematite.

thermodynamics, goethite and hematite are both more stable than ferrihydrate and often occur together as common transformation products of ferrihydrate (Cornell and Schwertmann, 2003). Estimates of the proportion of the three identified mineral phases are shown in Table 1. These were based on the assumption that Fe_o represents ferrihydrate (adjusted for stoichiometry) with the balance being goethite and hematite, which were estimated from peak heights and areas in the X-ray diffraction scans. The proportion of goethite and hematite that form via transformation of ferrihydrate is af-

ected by both pH and temperature, with warm temperatures and circumneutral pH favoring hematite over goethite. Thus, cooler storage temperatures (20°C) favor goethite formation while the initial pH of 7 to 8 favors hematite formation (Cornell and Schwertmann, 2003).

Mineral Transformations Induced by pH and Storage Time

In the experiment examining titration pH and storage time, iron oxides formed from the chloride and nitrate

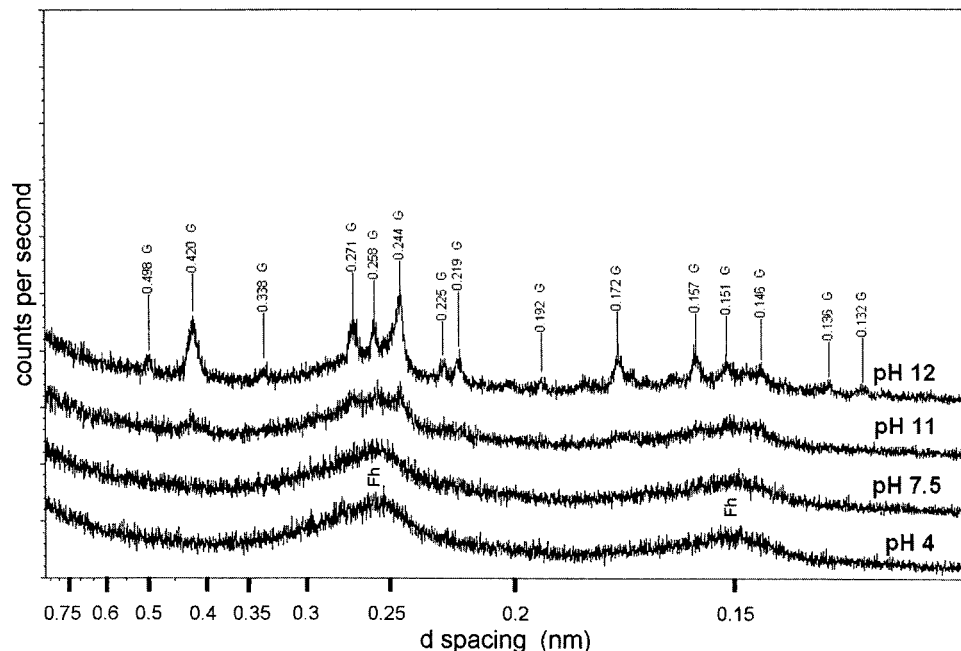


Fig. 5. X-ray diffractograms of synthetic Fe oxides 4 d old, formed by titration of Fe(III) chloride with KOH to a pH value of 4, 7.5, 11, and 12. Note two broad peaks for ferrihydrate (Fh) at 0.26 and 0.15 nm, and goethite (G) peaks are more pronounced in iron oxides formed at higher pH.

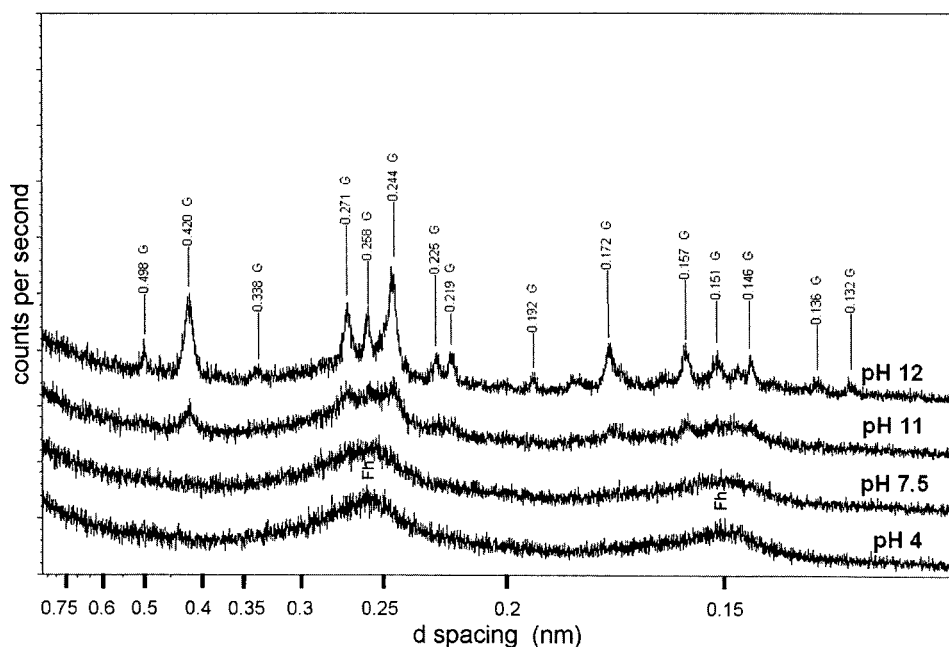


Fig. 6. X-ray diffractograms of synthetic Fe oxides 7 d old, formed by titration of Fe(III) chloride with KOH to a pH value of 4, 7.5, 11, and 12. Note two broad peaks for ferrihydrite (Fh) at 0.26 and 0.15 nm and goethite (G) peaks.

salts appeared to be nearly identical. Figure 5 shows the X-ray diffractograms for the iron oxides formed by titrating to pH values between 4 and 12, immediately after dialysis was completed, which was 4 d following their initial precipitation. Iron oxides formed at pH 4 and 7.5 show the two broad peaks characteristic of 2-line ferrihydrite. The formation of goethite was apparently favored by the titration to higher pH values of 11 and then to 12 (as shown by stronger goethite peaks). As the iron oxide suspensions age to 7 and 14 d, those

titrated to pH 4 and to pH 7.5 show no evidence of changing to more crystalline phases, but those titrated to pH 11 and to pH 12 show an increased intensity of the goethite peaks (Fig. 6 and 7). This trend appears to continue through a period of at least 42 d. X-ray diffractograms of iron oxides titrated to pH 4 and pH 7.5 show no evidence of goethite formation, while those formed by titrating to pH 11 or to pH 12 show evidence of continually increasing alteration of ferrihydrite to form goethite (Fig. 8, 9, and 10.)

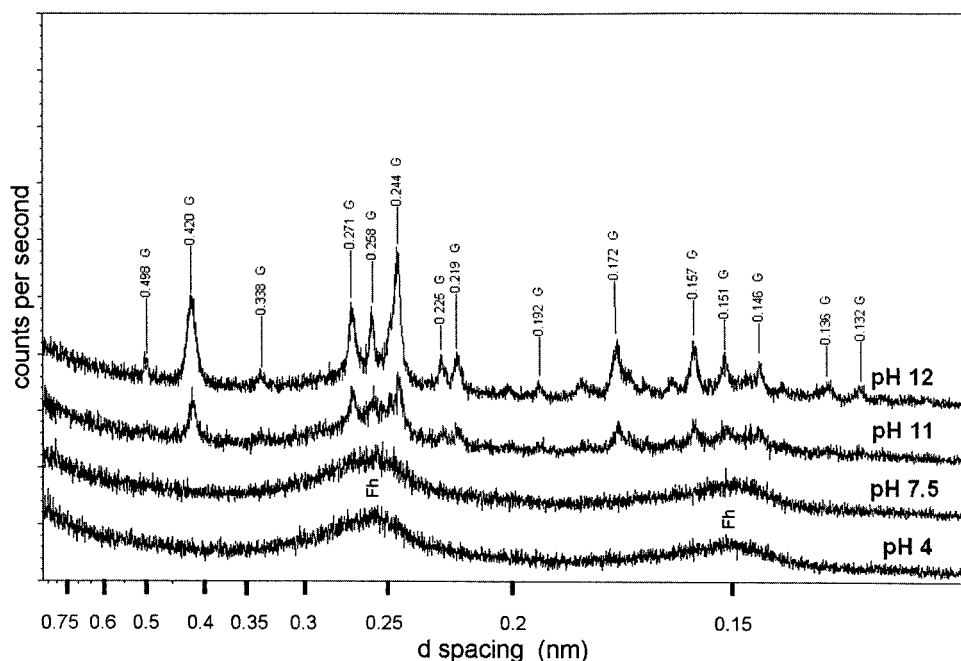


Fig. 7. X-ray diffractograms of synthetic Fe oxides 14 d old, formed by titration of Fe(III) chloride with KOH to a pH value of 4, 7.5, 11, and 12. Note two broad peaks for ferrihydrite (Fh) at 0.26 and 0.15 nm and goethite (G) peaks.

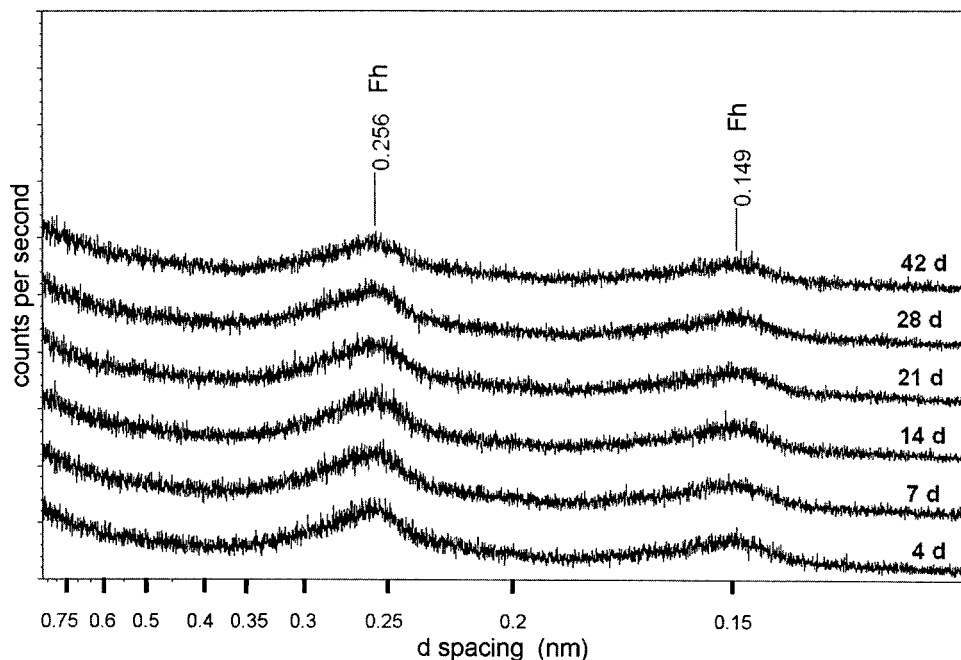


Fig. 8. X-ray diffractograms of synthetic Fe oxides of various ages (4 to 42 d), formed by titration of Fe(III) chloride with KOH to a pH value 7.5. Only two broad peaks for 2-line ferrihydrite (Fh) are observed.

The ratio of oxalate extractable iron to total iron (Fe/Fet) confirms more quantitatively what was observed qualitatively by X-ray diffraction. The iron oxide formed by titration to pH 4 and to pH 7.5 is entirely oxalate extractable, indicating that it is pure ferrihydrite (Fig. 11). When these materials are stored in an aqueous suspension, they remain largely unaltered as poorly crystalline ferrihydrite for at least 56 d. Other ferrihydrite samples synthesized in essentially the same manner have

remained in aqueous solutions for up to 80 d without showing alteration to goethite (data not presented). The iron oxides formed by titrating to pH 11 and pH 12 after 4 d are about 93 and 70% oxalate extractable, respectively (Fig. 11). The proportion of oxalate extractable iron continues to decrease logarithmically for these two materials so that after 56 d, the proportion of oxalate extractable iron is approximately 50 and 30%, respectively. This corresponds directly to the increase in the size of

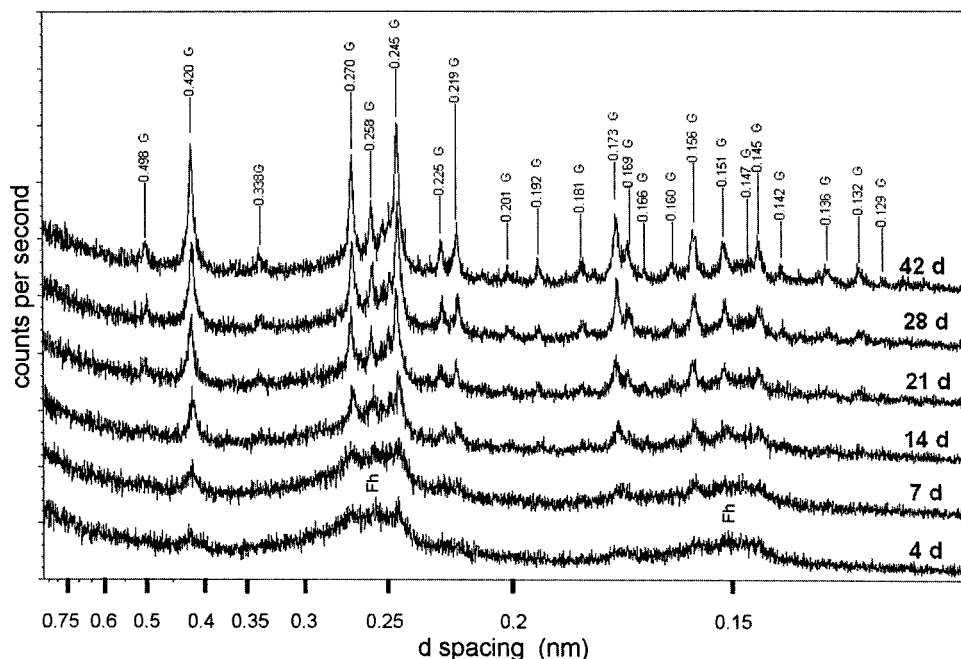


Fig. 9. X-ray diffractograms of synthetic Fe oxides of various ages (4 to 42 d), formed by titration of Fe(III) chloride with KOH to a pH value 11. Note goethite (G) peaks become more pronounced over time.

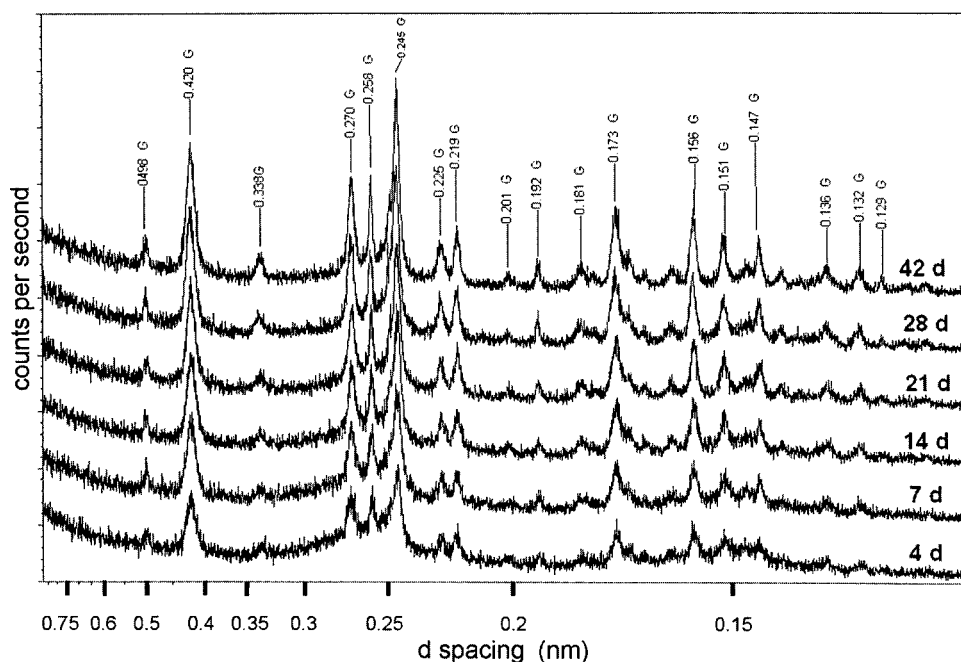


Fig. 10. X-ray diffractograms of synthetic Fe oxides of various ages (4 to 42 d), formed by titration of Fe(III) chloride with KOH to a pH value 12. Note goethite (G) peaks become more pronounced over time.

the goethite peaks in the X-ray diffraction patterns (Fig. 9 and 10).

Although Fe oxides were synthesized by titrating to a particular pH (such as 12.0), we expected that during the process of removing salts, the pH would likely change. To document this change, an additional sample of ferrihydrite formed by titrating to pH 12 was monitored for changes in pH associated with removal of salts over the course of several days following synthesis (Table 2). During the 3 d of centrifuge washing and dialysis, the pH decreased directly with the log of EC (Fig. 12) and then when dialysis was completed, appeared to stabilize at approximately pH 9.5. X-ray diffractograms illustrate

that approximately 1 to 2 d following synthesis, during dialysis, goethite begins to become visible (Fig. 13 and Table 2).

Durability of Iron Oxide Paint on IRIS Tubes

When the various suspensions of iron oxides were applied to PVC tubing in the construction of IRIS tubes, their suitability varied widely. Some of the paints (iron oxide suspensions) adhered well to the tubing and were resistant to abrasion, while others were easily rubbed off during normal handling. Each of the paints were evaluated and rated by a scale of 1 to 5 as explained

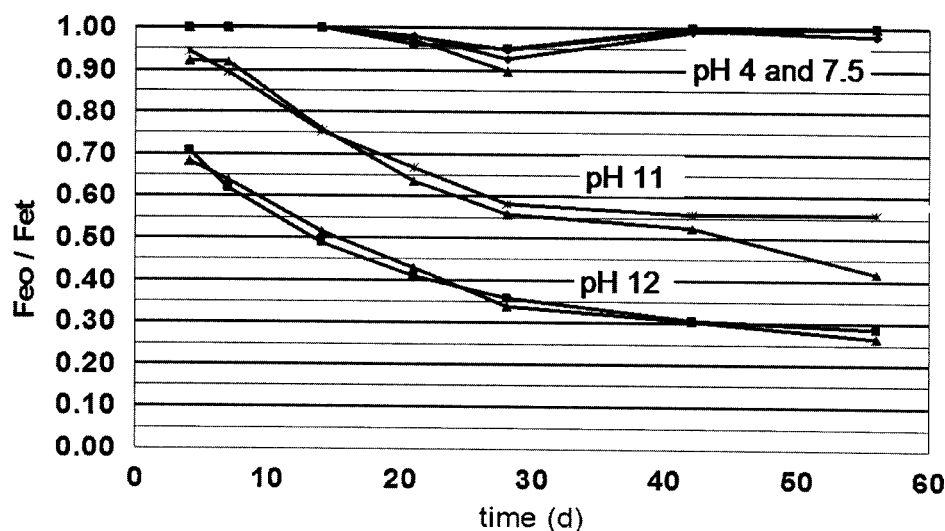


Fig. 11. Ratio of oxalate extractable Fe (Fe_o) to total extractable Fe (Fe_t) in synthetic Fe oxides formed by titrating to four pH values (4, 7.5, 11, and 12) and stored as aqueous suspensions in the dark over a period of 56 d. Note similarity of Fe_o/Fe_t for iron oxides synthesized at pH 4 and pH 7.5 and that the Fe_o/Fe_t ratio is very near to 1.0. Pairs of lines represent oxides synthesized from chloride and nitrate salts.

Table 2. Electrical conductivity (EC) and pH over time during salt removal in a ferrihydrite suspension synthesized by titrating to an initial pH of 12.0.

Description of process before sampling or measurement	XRD ID†	Date	Time		Suspension pH	EC
			h	d		
Initiation of titration		27 April 2005	0900			
Completion of titration		27 April 2005	0930	0.00	12.00	40.5
Centrifuge and resuspend 1		27 April 2005	1033	0.04	11.75	14.7
Centrifuge and resuspend 2		27 April 2005	1130	0.08	11.52	4.26
Centrifuge and resuspend 3		27 April 2005	1200	0.10	11.39	1.78
After Dialysis 1	a	27 April 2005	1630	0.29	11.13	0.82
After Dialysis 2	b	27 April 2005	2300	0.56	10.78	0.33
After Dialysis 3	c	28 April 2005	2330	1.08	10.54	0.16
After Dialysis 4	d	29 April 2005	1730	2.33	10.00	0.10
After Dialysis 5	e	30 April 2005	2030	3.46	9.61	0.04
Stored 2 d after Dialysis 5	f	2 May 2005	1500	5.23	9.62	nd‡
Stored 5 d after Dialysis 5	g	5 May 2005	0930	8.00	9.53	nd
Stored 7 d after Dialysis 5	h	7 May 2005	0930	10.00	nd	nd

† ID corresponds to scans of X-ray diffractograms shown in Fig. 13.

‡ Not determined.

earlier. These data are presented in Fig. 14, where the X axis approximates the proportion of goethite (non-oxalate extractable) in the sample. It appears that in order for the iron oxide paint to have good adhesion properties, it must contain a minimum of approximately 30 to 40% goethite. This corresponds fairly well with Castenson (2004), who reported that the paint she used for the construction of IRIS tubes contained approximately 30% iron oxides that were not oxalate extractable. Four additional paint samples that had been synthesized in our lab by titrating to pH 7.5, but not previously mentioned in this study, ranged in age from 61 to 82 d; they were evaluated and found to have poor durability with values between 1 and 2.

Schwertmann and Taylor (1989) have shown that ferrihydrite is reduced more easily than goethite or hematite. Thus ferrihydrite may more closely approximate newly formed iron oxides in the soil, especially in wetland soils that often pass through alternately reducing and oxidizing conditions. For this reason, ferrihydrite is a good choice for use in the construction of IRIS tubes

for assessing reducing conditions in soils. Because at least 30% goethite is required in the iron oxide paint to ensure proper adhesion to the PVC tubing, the transformation of ferrihydrite to more crystalline iron oxides during storage of the paint is to some degree necessary. This mineralogical transformation also represents a potential difficulty and source of error if too much of the ferrihydrite is transformed to goethite or hematite. IRIS tubes made using newly formed paint comprised primarily (60–70%) of ferrihydrite may be expected to perform differently from IRIS tubes made with older paint comprised mainly of goethite or hematite.

CONCLUSIONS

We conclude that the procedure outlined by Schwertmann and Cornell (2000) for synthesizing ferrihydrite (titrating to pH 7.5) is a good one and that the ferrihydrite made in this manner may remain stable for extended periods of time, even when stored as an aqueous suspension. A suspension of pure ferrihydrite, how-

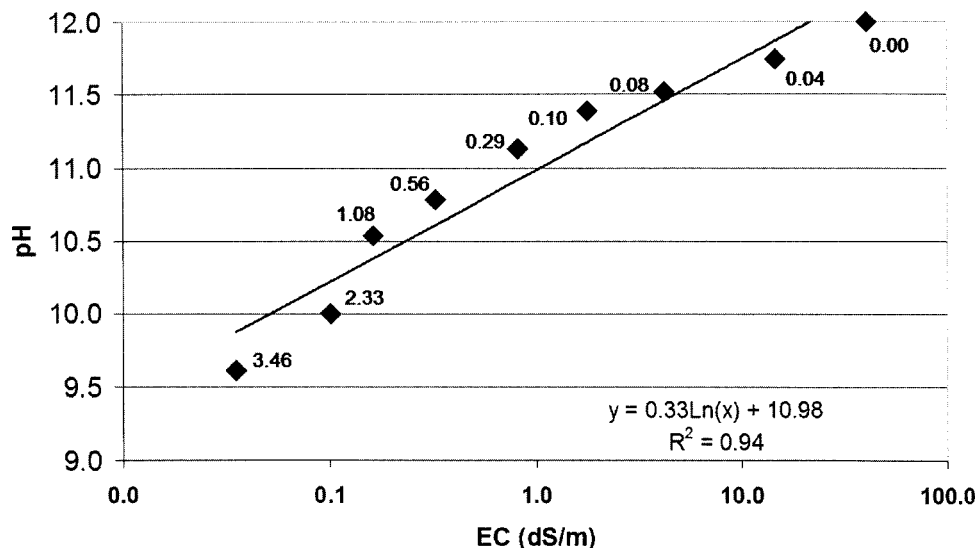


Fig. 12. Change in pH of synthetic Fe oxide suspensions (formed by titration to pH 12) as affected by removal of salts by centrifuge washing and dialysis over a period of three and a half days. Values by points are time (d) following original synthesis.

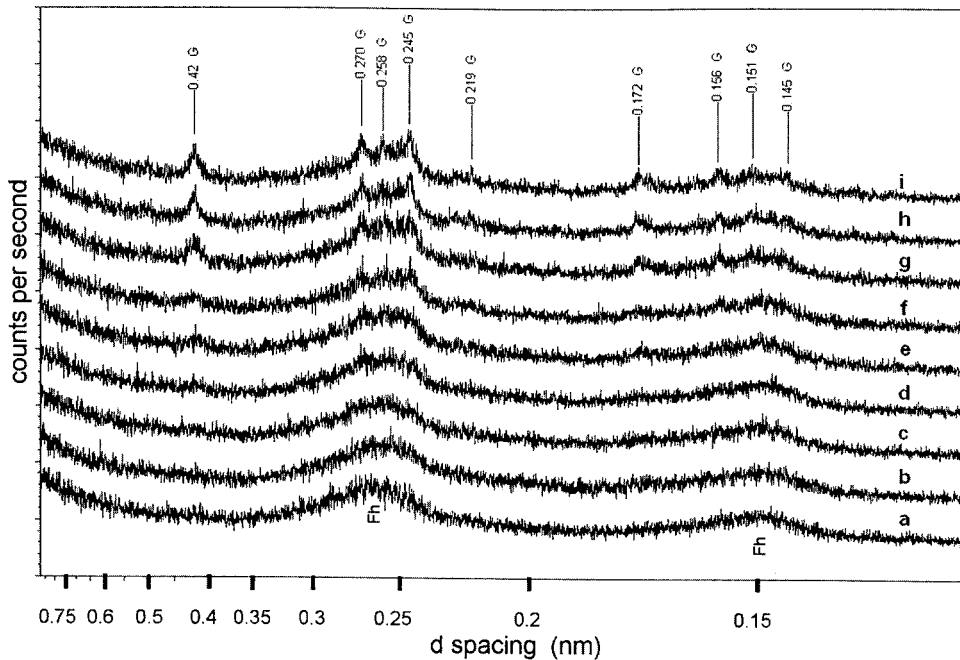


Fig. 13. X-ray diffractograms of synthetic Fe oxides sampled over a three and a half day period while salts were being removed by centrifuge washing and dialysis. Letters correspond to column 2 in Table 2. Goethite (G) peaks first begin to become visible in XRD scans d and e. Broad ferrihydrate (Fh) peaks are evident at 0.26 and 0.15 nm.

ever, does not appear to be a good material for coating IRIS tubes because of its poor durability and poor resistance to abrasion. In order for Fe oxide paint to adhere well to the PVC tubing, a minimum of 30 to 40% goethite must be present with the ferrihydrate. This mineralogical composition can be achieved by titrating the initial ferrihydrate suspension to pH 11 and allowing approximately 3 wk following initial synthesis for ferrihydrate transformation to goethite. Alternatively, if the ferrihydrate suspension is titrated to pH 12, the necessary mineralogical transformation will occur in approximately 1 wk. Because it is desirable that ferrihydrate

remain a dominant phase in the IRIS paint, and because mineralogical alterations of ferrihydrate to goethite continue over time, these paints will likely have a limited shelf life. On the basis of data present in this paper (Fig. 11), paint formed by titrating to pH 12 will probably have a relatively short useful shelf life of 1 to 2 wk. After the required aging period of approximately 3 wk, paint formed by titrating to pH 11 will apparently have a substantially longer shelf life of an additional 3 to 4 wk before goethite begins to become the dominant Fe oxide phase present. Paint that is synthesized by titrating to pH 7.5 will remain as nearly pure ferrihydrate and will

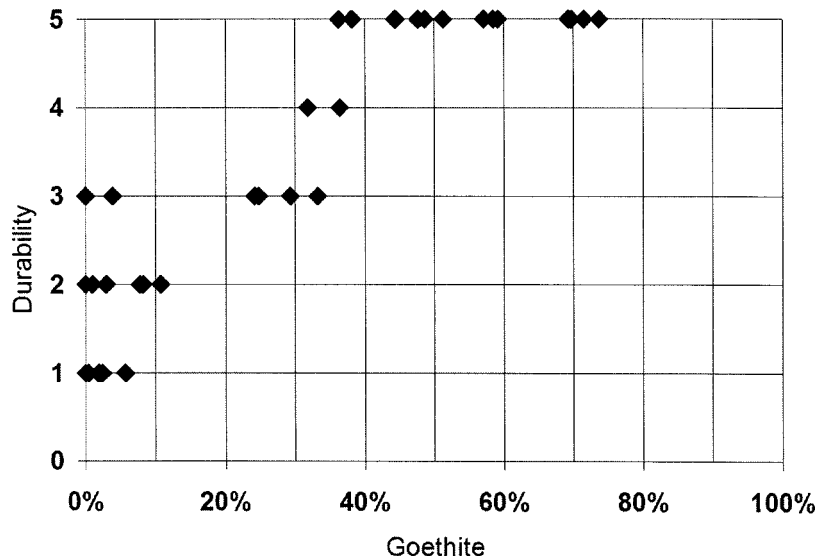


Fig. 14. Durability of Fe oxide paint when applied to IRIS tubes as a function of goethite content. A minimum of 30 to 40% goethite is necessary for painted tubes to have a durability index of five.

not have adequate adhesion and durability for use in constructing IRIS tubes.

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