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Iron Speciation in Selected Agricultural Soils of Peninsular Malaysia

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ABSTRACT

The study discusses iron speciation in upland and lowland agricultural soils of Peninsular Malaysia. Generally, iron speciation in upland soils is influenced by the formation of iron oxyhydroxides and iron precipitates, due to intense chemical weathering processes. The bioavailability of iron (easily leacheable and exchangeable, ELFE fraction) is very low and this could be attributed to its occurrence in the insoluble form. In the lowland soils, however, iron speciation varied widely due to varied soil composition, redox conditions and agricultural practices. Generally, soils in lowland regions are more clayey and silty compared to those in upland regions. The iron speciation in the Organic Oxizable (OO) fraction is also higher, due to the higher organic carbon content in soils. As with upland soils, the biovailability of iron in the lowland soil is also very low. In riverine alluvial deposits, iron tends to accumulate in the RR fraction, followed by the OO and Acid Reducible (AR) fractions. In the non-paddy areas, soils in the lowland region were found to be highly oxidized. Due to the shallow water table, these soils were exposed to the alternate submergence and exposure cycles during the wet and dry seasons respectively, resulting in the formation of iron and manganese mottles. Iron in these soils is concentrated mainly in the resistant fraction (RR), followed by the OO fraction. Due to the high organic carbon content, iron in peat soils is fixed mainly by organic matter (OO fraction) but it also existed in the resistant form (RR fraction).

Key words: Iron speciation, agricultural soils, Peninsular Malaysia

INTRODUCTION

Heavy metals occur naturally in rocks, soils, sediments and water but their anthropogenic components have increased significantly since the Industrial Revolution. This increase has caused serious environmental problems that have affected the food chain and consequently the health of organisms, including humans (Forstner, 1990). High levels of metals are usually found in superficial soil and vegetation of areas affected by anthropogenic activities such as mining, agriculture, metal industries and traffic emissions (Maiz *et al.*, 2000). Metals from antropogenic sources tend to be more mobile than pedogenic or lithogenic ones (Chlopecka *et al.*, 1996). It is a well established fact that metals are present in soils in forms that reflect their solubility and availability to plants (Zhang *et al.*, 1997), which influence their mobility and bioavailabilty (Abollino *et al.*, 2002). Some metals such as Cu, Zn, Fe and Mn are nutrients required by plants for various metabolic processes. Previous studies have indicated that if metal concentration exceeded the required amount, it may have toxic effects on the plants. However, the uptake of heavy metals by plants is influenced by various factors such as soil pH, organic matter content and clay content (Eriksson, 1990).

According to Lund (1990), the concentrations of heavy metals in soils are associated with biological and geochemical cycles and are influenced by anthropogenic activities. The knowledge of both the total concentration and chemical speciation of heavy metals is necessary to characterize their behaviour in soils. Understanding the distribution of various forms of metals in soils will provide insight into their solubility, transformation, chemical reaction and the pool of the plant available fraction besides helping in determining the judicious application rate of chemical and sewage sludge to agricultural soils (Zhang *et al*., 1997).

Iron is required in relatively large amounts by plants (Hopkins, 1999). It is taken up as a macronutrient, as both ferric (Fe^{3+}) or ferrous (Fe^{2+}) ion, although the latter is more common due to its greater solubility. Iron is also important as part of the catalytic group for many redox enzymes and is required for chlorophyll synthesis. Iron deficiency invariably leads to a simultaneous loss of chlorophyll and degeneration of the chloroplast structure (Hopkins, 1999). According to research carried out on iron stress by Connolly and Guerinot (2002), plants are able to adapt to excess iron content by storing it in the specialized iron-storage protein, ferritin.

Iron is the ubiquitos heavy metal in soils. It plays an important role in the behaviour of several trace elements. The mobilization and fixation of iron is influenced by the redox condition and the acidity of soils. Oxidizing and alkaline conditions promote the precipitation of iron, hence immobilizing the metal. On the other hand, acidic and reducing conditions promote the dissolving of iron compounds, thus releasing and mobilizing the metal (Kabata-Pendias and Pendias, 1984; Kirk, 2004). Mobility of iron into the secondary form from bedded sedimentary soils has been reported (Kadderi *et al*., 1990).

In Peninsular Malaysia, most of the regions are made up of igneous rocks as well as the older bed rocks of the Mesozoic and Paleozoic eras (Gobbett and Hutchison, 1973). Due to intense weathering, the bedrocks have developed into leached ferralitic soils which are acidic (Young, 1980) and with pH values usually below 5.5 (Jusop and Ishak, 2010). However, agricultural management practices such as liming and/or fertilizer application over decades have been capable of raising the pH of the topsoil (Jusop and Ishak, 2010).

Agricultural activities in Peninsular Malaysia, occur both in the highland and lowland regions. These areas represent highly oxidized soils with more complex soil conditions. The upland region includes mountain ranges and the lower areas of undulating hills, the eroded soils near cliffs and the areas that experienced leaching, eluviation and illuviation (Jusop, 2006).

The lowland region of Peninsular Malaysia includes riverine and marine alluvial deposits, peat and large flat areas of alluvium and peat swamps, extending inland along the western and eastern coast of Peninsular Malaysia. Apart from the peat deposits further inland, the alluvial plain is underlain by clay, silt and sand of the Pleistocene and Holocene ages. The alluvial deposits formed at various depositional environments include the coastal, offshore, inshore, mangrove and terrestrial environments as well as the peat deposits at paludal environments (Bosch, 1988). Due to the shallow water table, some areas have been exposed to seasonal water table fluctuation during the rainy and dry seasons. In addition, the paddy cultivation practices caused further exposure of these soils to the submerged and exposed conditions rendering the redox condition (Kyuma, 2004).

Understanding iron speciation in both the upland and lowland regions is crucial for agricultural management in Malaysia. It gives insight into iron related bioavailability and deficiency of soils under humid tropical conditions. This study attempts to describe the speciation of iron in agricultural soils of Peninsular Malaysia, with specific focus on iron concentration in the easily leacheable and iron exchangeable (ELFE), Acid Reducible (AR), Organic Oxidizable (OO) and resistant (RR) fractions.

MATERIALS AND METHODS

Several heavy metal speciation techniques have been used by a number of researchers. However, the present study only utilises iron speciation in soils following the technique developed by Badri and Aston (1983). The method was originally designed to investigate heavy metal pollution in estuarine sediments (Badri and Aston, 1983). Subsequently, it was successfully used for the study of heavy metal pollution in urban ecosystems (Badri, 1986; Badri, 1987; Badri, 1988; Badri and Rosnah, 1988), aquatic environments (Badri, 1990; Jusoh, 1987) and agricultural soils of Malaysia (Khairiah, 1994; Khairiah *et al*., 2006; Khairiah *et al*., 2009a; Khairiah *et al*., 2009b; Khairiah *et al*., 2009c; Jamil *et al*., 2011). In Peninsular Malaysia, iron speciation has been reported together with that of other heavy metals in various types of soils and crops including paddy, vegetables and fruits.

The above method is being used to quantify heavy metal concentrations in four geochemical phases, namely the easily leacheable and iron exchangeable (ELFE), Acid Reducible (AR), Organic Oxidizable (OO) and resistant (RR) fractions. The ELFE fraction accounts for the metals adsorbed to the negatively charged surfaces of clay minerals or organic matter and those easily soluble (Badri, 1988). Higher levels in the ELFE fraction indicate anthropogenic inputs in agricultural soils such as the impurities from fertilizers and pesticides (Khairiah *et al*., 2009b). The AR fraction accounts for the metals trapped within the stable Mn and Fe oxides, hydroxides and carbonates, which might dissolve under reducing conditions (Badri, 1988). Iron content in the AR phase might be higher in waterlogged agricultural areas especially paddy fields. The OO fraction contains the heavy metals associated with soil organic matter. The RR fraction represents the natural (non-anthropogenic) fraction of sediments, containing metals strongly incorporated into the crystalline lattice positions of minerals (Badri, 1988).

Single and sequential extraction procedures have been reported where different types of extraction procedures to extract metals were used but the most widely used procedure is that proposed by Tessier *et al*. (1979). This method was originally developed for sediment extraction but was later used to extract metals in agricutural and contaminated soils. In the present study, metals were extracted using a modified version of the sequential extraction method of Badri and Aston (1983). This method involves the use of a succession of chemical reagents chosen to selectively dissolve the various chemical or organic phases (Howard and Shu, 1996).

According to Sposito *et al*. (1982), the sequential extraction procedure is used to partition the total amount of metals into exchangeable, sorbed, organically bound, precipitated and residual forms. The technique was developed to predict the fate, transport and bioavailability of these elements in the soil system (Iskandar and Kirkham, 2001). However, only soluble, exchangeable and chelated metal species in the soils are the labile fractions available for plants (Kabata-Pendias, 1993). Heavy metals in these forms could be taken up by plants, bioaccumulated and eventually consumed by human beings. An excess of heavy metals in human bodies gives rise to several complications and illnesses.

Assessment of iron extracted from the various fractions using the above mentioned sequential extraction method, was carried out at selected agricultural areas in upland and lowland regions of Peninsular Malaysia, spanning different states and having different types of agricultural activities such as paddy, vegetable and fruit (guava) cultivation.

The upland study areas included two states ie: Kajang, Cheras and Serdang in the state of Selangor and Cameron Highlands in the state of Pahang. The lowland region was represented by Arau in the state of Perlis, Bumbung Lima in Seberang Perai, Penang, Besut in Terengganu, Sitiawan in Perak and Bangi and Sepang in Selangor.

Similar to previous studies (Khairiah, 1994; Khairiah *et al.*, 2006; Khairiah *et al.*, 2009a; Khairiah *et al.*, 2009b; Jamil *et al.*, 2011), soil samples were collected randomly from the study areas at 0-30 cm depth. Soil samples were air-dried in the laboratory environment. The soil was passed through 250 μm mesh and later grounded using a mortar and pestle. Iron was extracted from the soil using the sequential extraction method according to Badri (1984) and its concentration was determined by AAS (atomic absorption spectrophotometry) Perkin Elmer (model 1100B). Other parameters studied were organic carbon (Walkley and Black, 1934), soil pH (Duddridge and Wainwright, 1981) and grain size (Badri, 1984).

RESULTS AND DISCUSSION

Total iron content in the agricultural soils: Total iron content in the agricultural soils of the upland and lowland regions are displayed in Fig. 1. Most of the agricultural practices in the upland regions were mainly confined to undulating hilly land and eroded soils near the foot hills (colluvium). The results indicated that the total iron concentration in the soils of the hilly land areas were higher than that of the colluvium soils. The iron concentration in the soils from both the highland vegetable farming areas in Cameron Highland and Cheras were 397.20 and 322.20 mg kg^{-1}, respectively.

The soils at Kajang and Serdang constituted unsorted colluvial deposits at the cutting slope [slope cut] and near the foothills. The total iron content for the Kajang soil was 42.30 mg kg^{-1}

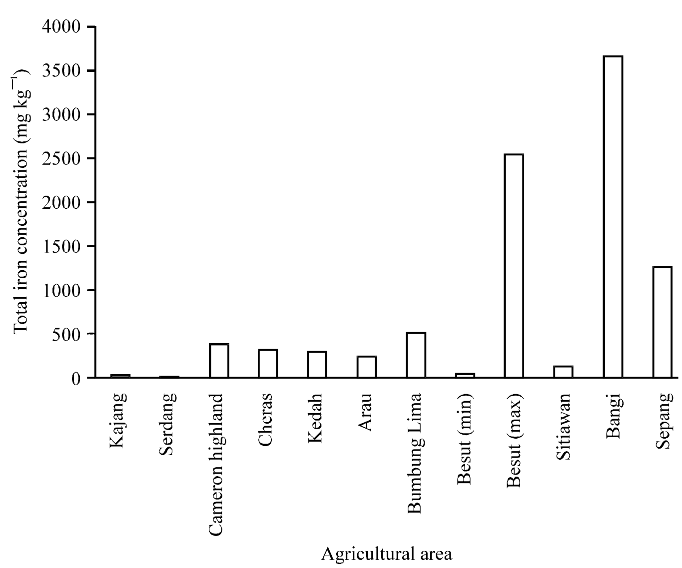

Fig. 1: Total iron concentration in agricultural soils of Peninsular Malaysia

whereas that of Serdang was 21.26 mg kg^{-1}. The low total iron concentration in these soils could be attributed to a lower amount of iron oxides in the pseudo-silt and pseudo-sand. The unsorted materials in the colluvial deposits constituted a mixture of iron oxides and oxyhydroxides, resistant primary minerals (quartz) and altered primary minerals. Soils in the area were also disturbed by levelling activities.

The properties of soils in the lowland regions varied widely depending on their parent materials (Kirk, 2004). In Peninsular Malaysia, the marine and riverine alluvium as well as the peat deposits consist of clay, silt, sand and organic matter. The marine alluvial deposits were formed from the raised sea bed levels during the Holocene period (Bosch, 1988). The riverine alluvial deposits constituted the channel, point bar, levee and floodplain areas, whereas peat accumulated in the swampy areas. The total iron content in soils was greatly affected by the soil composition.

Paddy cultivation in Kedah, Arau and Bumbung Lima was carried out on the soils developed from marine alluvial deposits. These soils contained higher amount of silt and clay (73.15-82.00%) compared to those of the upland regions (9.10-42.33%) and were slightly acidic (pH 5.12-5.62). The total iron concentration in the paddy soils of these areas did not differ much from those of the upland regions because these soils also contained high amounts of iron oxide. Soils developed from the riverine alluvial deposits were more heterogeneous. The paddy soils in Besut originated from young riverine alluvial deposits, whereby soils on the valley floors were partly colluvial in origin and were formed by sedimentation from flooding. In certain areas, localized depressions enabled the accumulation of organic matter rendering the soils rich in organic carbon content. Thus, the total iron concentration in the paddy soils of Besut varied widely due to the difference in soil formation.

The lowland regions are also used for vegetable and fruit cultivation. Guava plantations in Sitiawan are situated along the coastal plain, the soils of which constitute fluviatile deposits (Bosch, 1988). Vegetable farming has also been undertaken on soils developed from riverine deposits in inland valleys (Bangi). The total iron concentration in both lowland areas was markedly different. In Sitiawan, the total iron concentration was 132.51 mg kg^{-1} whereas in Bangi, its concentration was 3658.03 mg kg^{-1}. The soils of Bangi constituted a mixture of riverine alluvial deposits with inputs of eroded materials from the nearby hilly areas. The input of reddish colour earth materials from higher topographical areas indicate higher iron oxide content in the soils, which contributed to the high total iron concentration in the soils. The Sitiawan area is located ten kilometres from the hilly area suggesting that the sediments in the area were low in iron. Upon weathering, the Fe^{2+} and Fe^{3+} iron from the rock were released to the environment (Kampf *et al.*, 2000). Some were oxidized and occurred in the resistant form, whereas the dissolved fraction was transported to the lower topography via the fluvial system or seeped through the subsurface and transported in groundwater. Consequently, the dissolved elements would precipitate and accumulate in the sediments of the depositional basin, should favourable conditions occur. Along the pathway, most of the iron would be precipitated and immobilized, rendering a lower amount of iron in the soil further from the sediment source. This situation explains the lower iron concentration in the soils of Sitiawan compared to those of Bangi. The agricultural soils of Sepang were acidic (pH 4.52), rich in organic carbon (26.85%) and contained lower amounts of particles with grain size of less than 63 μm (58.26%). The area was covered with peat, humic clay and silt of Holocene paludal deposits (Bosch, 1988) and the total iron concentration was high.

Iron speciation in the upland agricultural soils: The iron speciation data and soil properties of the upland agricultural soils are displayed in Table 1 and Fig. 2. Soils from the Cameron

Table 1: Iron speciation, pH values, organic carbon content and grain size (less than 63 μm) in selected agricultural soils of Peninsular Malaysia

Agricultural area	Iron speciation (mg kg^{-1}) RR	OO	RA	ELFE	Total	pH value	Organic carbon content (%)	Grain size less than 63 μm (%)	Sources
Upland region									
Kajang	10.27	4.36	26.01	1.66	42.30	6.57	1.28	15.72	Current study
Serdang	11.39	5.08	3.22	1.57	21.26	4.17	0.13	31.28	Current Study
Cameron highland	344.53	39.67	12.30	0.70	397.20	6.75	2.23	42.33	Khairiah *et al*. (2006)
Cheras	314.80	5.90	0.50	1.00	322.20	7.23	2.43	9.10	Khairiah *et al*. (2006)
Lowland region									
Kedah	72.60	137.54	99.59	2.11	311.84	4.94	5.67	80.82	Khairiah *et al*. (2009c)
Arau	30.36	128.28	95.65	0.62	254.91	4.63	5.53	73.15	Khairiah *et al*. (2009c)
Bumbung Lima	181.340	209.975	128.671	0.423	520.409	6.54	5.12	82.00	Jamil *et al*. (2011)
Besut	409.827- 2133.38	133.00- 466.460	57.77- 101.39	0.456- 1.848	859.35- 2552.90	5.45- 6.12	1.46- 9.86	67.37- 76.82	Khairiah *et al*. (2012)
Sitiawan	91.91	28.00	12.60	0.00	132.511	4.76	1.42	64.50	Khairiah *et al*. (2009a)
Bangi	3607.83	41.26	8.50	0.44	3658.03	6.83	1.24	17.54	Khairiah *et al*. (2009b)
Sepang	611.98	659.55	3.87	1.24	1276.64	4.52	26.85	58.26	Khairiah *et al*. (2009b)

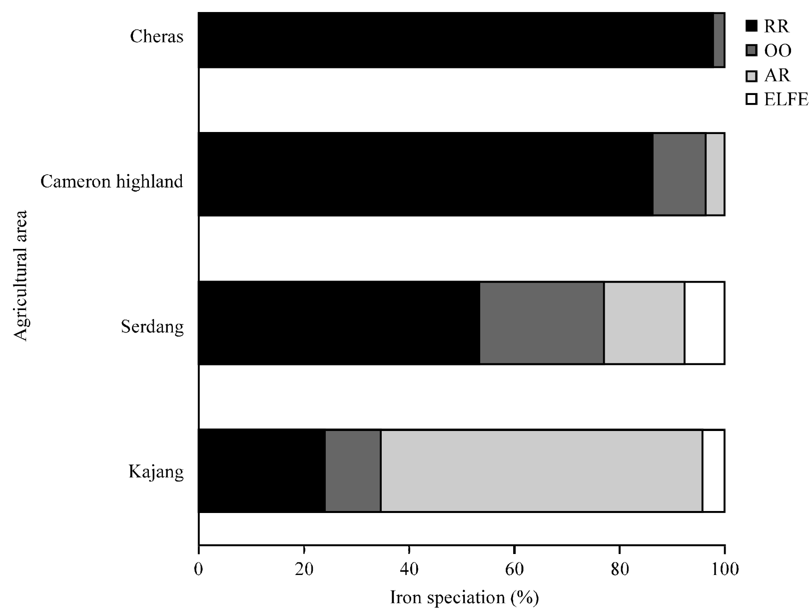

Fig. 2: Iron speciation in upland agricultural soils

Highlands and Cheras were developed from weathered metasediment and granitic rocks and were rich in iron in the RR fraction which accounted for more than 85% of the total iron content in the said soils. Iron speciation in the hilly land could have occurred by the formation of iron oxyhydroxide precipitates and minerals in the soils due to intense chemical weathering of rocks. In humid tropical regions such as Malaysia, rock weathering gives rise to the formation of ultisols and oxisols. According to Jusop and Ishak (2010), the clay fraction in the ultisols of Peninsular Malaysia is dominated by kaolinite and the oxides of Fe and Al. They are often rich in Fe and Al oxide minerals. By comparison, the oxisols contain high amounts of sesquioxides, hematite (Fe_2O_3) and goethite (α-FeOOH). The clays are cemented by the oxides of Fe to form pseudo-sand and pseudo-silt. The formation of pseudo-silt and pseudo-sand is common in the oxisols and this could be attributed to the accumulation of iron precipitates and minerals into crumbly nodules. In the present study, the fixation of iron in pseudo-silt and pseudo-sand rendered the soils in the upland region sandy and the iron occurred in the insoluble and resistant forms (RR fraction). As a consequence, the bioavailability of iron in these soils is low (ELFE fraction).

In the Kajang soil, more than half the iron concentration was found in the AR fraction followed by the RR, OO and ELFE fractions. For the Serdang soil, nearly half of the iron content is contained in the RR fraction, followed by the OO, AR and ELFE fractions. Higher amount of soil iron in the AR fraction could be explained by the occurrence of ferrihydrite ($Fe_5HO_8.4H_2O$) in the colluvial deposits. This poorly ordered Fe oxide, with a variable degree of ordering (Kampf *et al.*, 2000) might have entered into solution under reducing conditions (Kirk, 2004). Some of the iron is also trapped into the more stable and insoluble iron oxides, as indicated by the high iron concentration in the RR fraction. Iron was present as Fe^{2+} in primary minerals (mainly silicates) of most rocks. Upon weathering, the Fe^{2+} is released to the aerobic environment. It is immediately oxidized to Fe^{3+} to form ferrihydrite or goethite. Individual hematite crystals then nucleated and grew within individual ferrihydrite aggregates by dehydration and rearrangement processes (Kampf *et al.*, 2000).

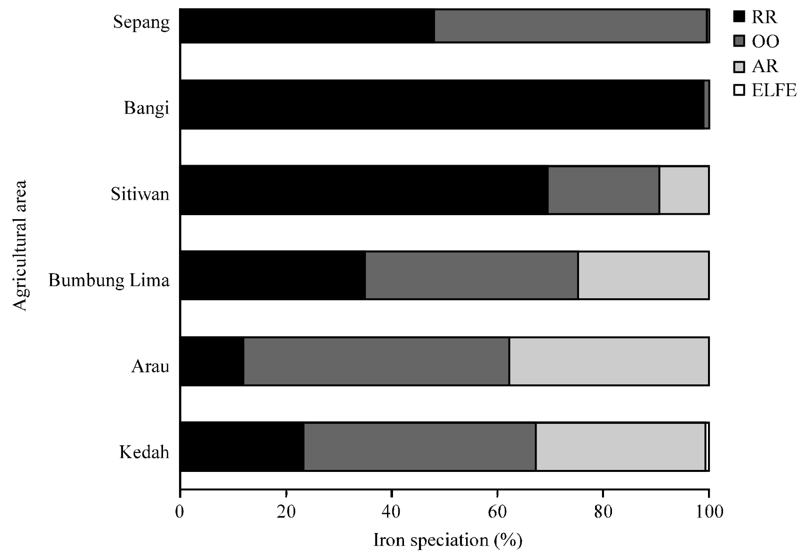

Fig. 3: Iron speciation in lowland agricultural soil

Iron speciation in the lowland agricultural soil: The iron speciation data and soil properties of the lowland agricultural soils are displayed in Table 1 and Fig. 3. As with the total iron concentration, the wide variation of the iron speciation in the lowland region was also signified by the soil composition. Apart from soil composition, iron speciation was also influenced by agricultural activities. For example, paddy cultivation was carried out in soils developed from both the marine and riverine alluvial deposits. Simultaneously, both types of the above mentioned soil types were also utilised for vegetable and fruit cultivation.

Iron in paddy soils developed from marine alluvial deposits (Kedah, Arau and Bumbung Lima) was concentrated in the OO fraction (128.28-209.98 mg kg^{-1}), which accounted for approximately 40-50% of the total iron content in the soils. This was followed by the AR (95.65-128.67 mg kg^{-1}) and RR (30.36-181.34%) fractions. The concentration of iron in the ELFE was very low (0.42-2.11 mg kg^{-1}), amounting to less than 1% of the total iron content. It is widely accepted that organic matter plays an important role in heavy metal fixation in soils. The organic carbon content in the paddy soils of Peninsular Malaysia was fairly high (4.83-6.54%), corresponding to the high concentration of iron in the OO fraction. The high iron concentration in the AR and RR fractions could be explained by the occurrence of iron precipitates and mottles in paddy soils due to the submergence and emergence cycles in the paddy field throughout cultivation and harvesting activities. The alternate flooding and drying conditions lead to a redox condition, which in turn results in the formation of ferrihydrite and goethite (Manceau *et al*., 2005). According to Kawaguchi and Kyuma (1969), paddy soils in Malaysia experience high intensity oxidative processes during the dry season, normally after harvesting and goethite (α-FeOOH) was commonly found in paddy soils of tropical Asia (Kirk, 2004) and in the subsoils of Malaysian paddy soils (Kawaguchi and Kyuma, 1969). In a reduced environment, goethite and ferrihydrite are formed by abiotic or biotic processes. It is also interesting to note that the biogenic or bacterial origin of inorganic, magnetite has been reported (Mann *et al*., 1984). The presence of ferrihydrite together with goethite might indicate that environmental conditions were not favourable for crystal development (Kampf *et al*., 2000). Thus, besides organic chelation, iron accumulates as the iron precipitates and mottles.

In paddy soils developed from young riverine alluvial deposits (Besut), iron was highly concentrated mainly in the RR fraction, followed by the OO fraction. The iron concentration in the AR and ELFE fractions was low. Mottles of iron oxide are commonly found in paddy soils of the east coast of Peninsular Malaysia (including Besut). It was reported that paddy soils of the east coast of Peninsular Malaysia were significantly higher in Fe than those of the west coast (Kawaguchi and Kyuma, 1969; Paramananthan, 1989). The occurrence of iron oxide mottles explained the high iron concentration in the resistant RR fraction. The organic carbon content in the Besut soils ranged from 1.46 to 9.86%. The higher amount of organic matter increased the fixation of iron in the soils.

Most of the soil iron in the non-paddy cultivation areas (Bangi and Sitiawan) was concentrated in the RR fraction, which amounted to approximately 98 and 69%, respectively, followed by the OO and AR fractions. The bioavailable iron was very low, as indicated by the low iron concentration in the ELFE fraction. Soils in these areas were not subject to alternate flooding and drying cycles as in the case of paddy soils. Most of the time, the soils were exposed to aerated conditions. However, due to the shallow water table, the seasonal fluctuations in the water table resulted in the formation of iron oxide mottles. As such, iron tended to occur as resistant iron oxide mottles in the soils.

Most of the soil iron in peat deposits of Sepang area was concentrated in the OO (659.55 mg kg^{-1}) and RR (611.98 mg kg^{-1}) fractions, which amounted to 51.66 and 47.94% of the total iron content in the soils, respectively. The concentration of iron in the AR and ELFE was very low. In rich organic and peat soils, iron tends to accumulate in organic matter, as indicated by the high iron content in the OO fraction. This was due to the high cation exchange capacity, dominated by hydrogen ions (Isahak, 1992). Iron was also fixed in the resistant form (RR fraction).

CONCLUSION

Iron speciation in agricultural soils of Peninsular Malaysia reflects the soils composition, redox condition and agricultural activities. In the upland region, iron was mainly concentrated in the resistant iron oxide precipitates and minerals (RR fraction) at the top soils. Due to the unsorted natured soil mixture at the foothill and in the riverine alluvial deposits, iron speciation was widely varied and less understood. In the lowland region, iron was more concentrated in the OO fraction due to the higher organic carbon content in the soils of the lowland region compared to those of the upland region. Due to the high content of organic carbon, the iron in peat soils was fixed by the organic matter. Besides the OO fraction, iron in the paddy soils was also high in the AR fraction (compared to that of the non-paddy cultivated areas). The non-paddy area contained high iron content in the RR fraction, suggesting its occurence as mottles of iron oxide. In both regions, the bioavailability of iron in soils was very low or negligible.

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