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MINERAL PRECONCENTRATION USING NEAR INFRARED SENSOR-BASED SORTING

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Abstract: This paper predicts qualitatively and quantitatively the near infrared activity of individual minerals in simple and complex mineral associations using mixtures of common alteration minerals found in a copper ore. It was found that spectra dominance in most cases is dependent on any or combination of mineral composition, relative proportion or concentration and/or mineral accessibility or sensitivity to near infrared radiation. The analysis of results also indicated that, in most cases, only freely occurring waste, such as clays (kaolinite and/or muscovite) and calcite, can be targeted for discrimination. In this paper, a strategy for the application of near infrared for preconcentration of copper bearing minerals like chrysocolla and malachite from coarse ore particles was proposed. Other applications also considered in this paper include preconcentration of hematite from associated clays and carbonate waste, and the determination of moisture content in kaolinitic clays.

Keywords: *near infrared, copper, hematite, preconcentration, strategy, intimate mixtures, moisture*

Introduction

The main goal of near infrared (NIR) application in a mineral processing operation is to accurately classify the composition of individual spectra obtained by scanning and classifying ore particles according to their value. This is complicated by the fact that most spectra are influenced by a complex mixture of minerals. Hence, a number of minerals could exist within the range in varying compositions and arrangements among other modes of occurrence. In order to develop an efficient sensor-based sorting process, determination of the point of dominance of individual minerals and ratio at which spectral mixing is reached is important.

In the longer wavelength region of NIR (1300 to 2500 nm), a few functional groups (-OH, H₂O and or CO₃²⁻) dominate spectra due to vibrational processes (Clark, 1995). Based on the presence or absence of these functional groups, minerals can be classified into three major groups: a) NIR-active minerals which display absorption

features, b) NIR-active minerals which do not display absorption features, and c) non-active minerals. Note that NIR-active minerals without distinctive absorption features are highly absorbing throughout the longer wavelength region of NIR, with the ability of masking feature of other minerals (Iyakwari and Glass, 2014). Even when present in combination with other minerals, non-active minerals do not influence NIR spectra either by absorbing or displaying absorption features. In some instances the NIR features in non-active minerals are a result of either impurities (fluid inclusions) or external environmental interference (moisture) (Aines and Rossman, 1984; Pommerol and Schmitt, 2008; Iyakwari et al., 2013).

This paper aims to predict individual NIR-active minerals exerting a dominant influence over other mineral responses within the same NIR spectral range. Mineral associations as present in a hydrothermally-formed supergene-enriched copper deposit found in the Atacama district in Northern Chile (Daroch and Barton, 2011) are used in this paper. From the modal mineralogy, chrysocolla and malachite are the copper-bearing minerals in the ore. Both minerals are absorption features displaying NIR-active minerals (Iyakwari et al., 2013). Associated NIR-active waste minerals in the ore include kaolinite, calcite, muscovite, chlorite, biotite and hematite (Iyakwari et al., 2013; Iyakwari and Glass, 2014). Hence, chrysocolla and malachite preconcentration is scoped from these associated NIR-active waste minerals in the ore. Preconcentration of iron oxide ore (hematite) from clay and/or carbonate gangue minerals and the potential for using NIR for determination of moisture content of kaolinitic clays are also considered.

According to Clark et al. (1999), there are four types of complex mixtures or associations of materials: intimate, linear or areal, coatings, and molecular mixtures. With respect to current research, only intimate mixtures consisting of two or more minerals were investigated. This research aims to further study intimate mixtures of NIR-active minerals and explore strategies of applications in complex ore preconcentration.

According to Iyakwari and Glass (2014), the NIR feature depth and reflectance are affected by variation in the particle size. Hence, to avoid the influence of varying particle size, similar particle sizes were used in this study. Pure minerals were mixed with the sole aim of quantitatively and qualitatively classifying or defining them based on their best diagnostic absorption features in NIR (Iyakwari and Glass, 2014), and developing discrimination strategies from associated waste. This study is intended to reveal the degree of sensitivity of these minerals individually in the mixtures. The investigation also aims to establish whether minerals can be individually identified and classified relative to one another, and at what ratio spectra appear mixed, showing features relating to all minerals in concentration, or when one mineral relative to the other(s) becomes invisible (masked or replaced) in a spectrum.

Material and methods

Eight NIR-active minerals with expected association in a copper ore were selected: calcite and malachite (carbonate-bearing), kaolinite, muscovite, chrysocolla, chlorite (clinochlore), biotite (hydroxyl- and water-bearing) and hematite. Minerals were crushed and ground to $-45\ \mu\text{m}$ particle size fraction. The ground samples were first analysed for purity using X-Ray Diffraction (XRD, Siemens/Bruker D5000). XRD measurements were matched with known mineral signatures using EVA software. Results of XRD indicate that the samples were of high purity.

Table 1 presents NIR-active minerals in the ore, indicating their diagnostic feature(s) wavelength absorption position between 1300 and 2500 nm, and the functional groups responsible for their absorption. Ground minerals ($-45\ \mu\text{m}$) were intimately mixed according to prescribed ratios of mass. While simple mixtures involving two minerals were only prepared for a series of mass ratios, complex mixtures involving three or more minerals were only created with equal mass of each mineral. Each mixture was homogenised using a Grant-bio PTR-60 end-to-end shaker. The end-to-end shaker was set to rotate at 30 rpm for eighteen hours. Following this treatment, the mixtures appeared to be homogeneous. In order to achieve the desired objectives, the investigation was broken down into four parts:

- a) minerals with similar functional groups,
- b) minerals with dissimilar functional groups,
- c) influence of hematite on NIR-active feature displaying minerals,
- d) complex mixture or associations.

The spectra were generated by a NIR line scanner, which measures on a succession of adjacent areas across the mineral surface, each with a size of 2.9 mm by 9 mm. The measured NIR signal was converted to a reflectance by first measuring the upper and lower signal values. For the upper limit, denoted I_{light} , a highly-reflective board made of aluminium was scanned. For the lower limit, denoted I_{dark} , a scan was made in the absence of near infrared illumination. The NIR signal, I , was then converted into a reflectance, R , as follows (Iyakwari et al., 2013):

$$R = \frac{I - I_{\text{dark}}}{I_{\text{light}} - I_{\text{dark}}}. \quad (1)$$

Field data are often accompanied by noise self-generated by the sensor and/or the result of physical fluctuations in surrounding environment (Stark and Lutcher, 2005). All spectra were smoothed using OriginPro 9.0 software with the Savitzky–Golay method (Savitzky and Golay, 1964), applying a polynomial of order 2 to a frame size of 9 points.

Table 1. Absorption features of NIR-active minerals (between 1300 nm and 2500 nm) in copper ore (after Iyakwari et al., 2013; Iyakwari and Glass, 2014)

Group	Mineral	Molecule absorption feature, nm		
		-OH	H ₂ O	CO ₃ ²⁻
Silicate	chrysocolla	1415, 2270	1415, 1915	–
	muscovite	1415, 2205, 2350	1415, 1840, 1915	–
	kaolinite	1400, 1415, 2160, 2200	1415, 1840, 1915	–
	biotite	2255, 2370	1920	–
	chlorite	1415, 2265, 2360	–	–
Carbonate	calcite	–	–	1920, 2000, 2150, 2340
	malachite	2360	–	2275, 2360
Oxide	hematite	non-feature displaying NIR-active mineral		

Results and discussion

NIR spectra of intimate mixtures (Tables 2 and 3) revealed that when a particle contains mixtures of minerals, the visibility of absorption features of individual minerals in its NIR spectrum or spectra may depend on any or the combination of these mineralogical factors:

- 1) NIR-active mineralogical composition,
- 2) relative proportion or concentration, or
- 3) relative mineral accessibility or sensitivity to NIR radiation.

Other factors may include instrumental parameters, such as the spatial and spectral resolution (the narrowest spectral feature that can be resolved by a spectrometer) of the sensor, as well as the sensor sensitivity and mode of scanning. For some intimate mixtures, spectra dominance depends on concentration and mineral accessibility to NIR radiation. An example of concentration-dependent mixture (association) is chrysocolla and muscovite (Fig. 1 and Table 2). The mineral with higher concentration will dominate the spectrum of mixture. The mixture of muscovite and chlorite (Fig. 2 and Table 2) is a good example of mineral accessibility-dependent association. Here, muscovite dominates spectra even at lower concentration relative to chlorite, implying that muscovite is more readily accessible to NIR radiation than chlorite.

In other instances, spectral dominance depends on the NIR-active mineral composition, with minerals behaving differently in different mixtures. For example, weak mineral in one mixture may be strong when mixed with another mineral. An example is chlorite: the spectrum of both minerals mixed with chrysocolla (Fig. 3)

shows features of the minerals side-by-side. The visibility of features of either mineral is concentration-independent as both minerals are equally NIR-sensitive. In chrysocolla-malachite mixtures, chrysocolla shows more spectral dominance. This is not observed for chlorite mixed with malachite. When mixed with malachite, chlorite features are captured, meaning that malachite is more readily accessible to NIR than chlorite.

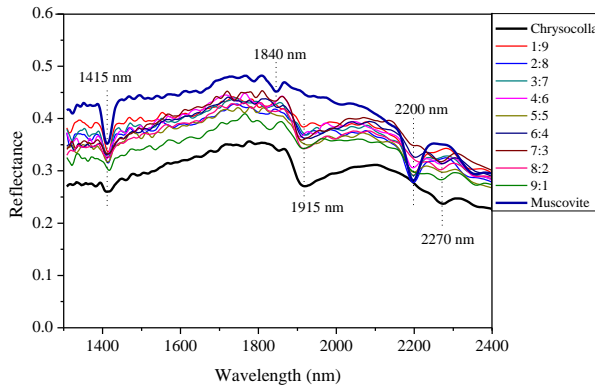


Fig. 1. NIR spectra of chrysocolla-muscovite mixtures. Bottom and top spectra are reference for chrysocolla and muscovite, respectively

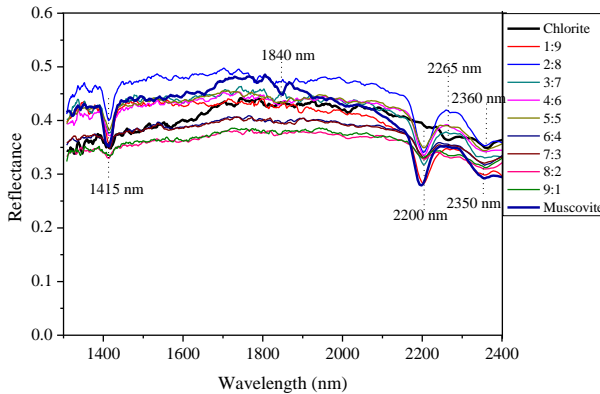


Fig. 2. NIR spectra of chlorite-muscovite mixtures. Bottom and top spectra are reference for chlorite and muscovite, respectively

Apart from the weakness of malachite in chrysocolla mixtures and the complete spectral mixing of malachite and kaolinite (Fig. 4), only the effect of malachite and hematite is fairly consistent across range of mixtures. The consistency of both minerals may be due to their colour (Clark, et al., 1999). Malachite may also have additional influence due to its $-OH$ and CO_3^{2-} combination chemical structure, making it strong enough to display its absorption feature (2275 nm) even at higher hematite concentration when both are mixed together (Fig. 5).

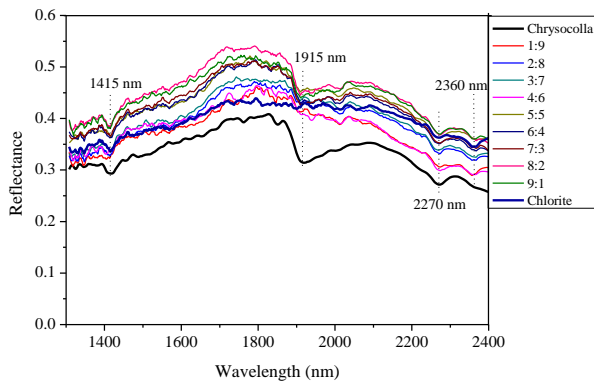


Fig. 3. NIR spectra of chrysocolla-chlorite mixtures. Bottom and top spectra are reference for chrysocolla and chlorite, respectively

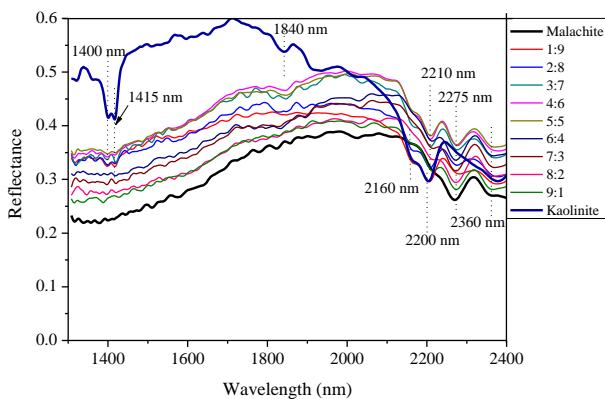


Fig. 4. NIR spectra of malachite-kaolinite mixtures. Bottom and top spectra are reference for malachite and kaolinite, respectively

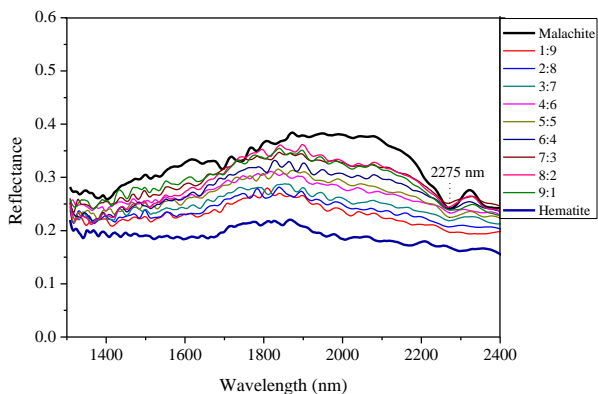


Fig. 5. NIR spectra of malachite-hematite mixtures. Bottom and top spectra are reference for hematite and malachite, respectively

Table 2. Spectral dominance in intimate mixture of minerals

Mineral 1	Mineral 2	Mass ratios of minerals (mineral 1: mineral 2)								
		1:9	2:8	3:7	4:6	5:5	6:4	7:3	8:2	9:1
Minerals with similar functional groups										
chrysocolla	muscovite	Muscovite			mixed spectra			chrysocolla		
chrysocolla	kaolinite	Kaolinite				mixed spectra				
chrysocolla	chlorite	mixed spectra								
chlorite	muscovite	muscovite								
kaolinite	muscovite	muscovite						kaolinite		
chrysocolla	biotite	featureless spectra								
kaolinite	chlorite	kaolinite								
chlorite	biotite	featureless spectra								
biotite	muscovite	muscovite								featureless spectra
malachite	calcite	malachite								
Minerals with dissimilar functional groups										
chrysocolla	calcite	broad spectra				chrysocolla				
muscovite	calcite	mixed spectra				displaced muscovite (2350 nm) features	muscovite			
kaolinite	calcite	mixed spectra				kaolinite				
chlorite	calcite	broad spectra			chlorite					
malachite	chlorite	malachite								
malachite	kaolinite	mixed spectra								
chrysocolla	malachite	mixed spectra				chrysocolla				
biotite	calcite	calcite								
malachite	biotite	broad spectra	malachite							
malachite	muscovite	mixed spectra				malachite				
Influence of hematite on NIR-active features displaying minerals										
chrysocolla	hematite	hematite (featureless spectra)								chryso-colla
malachite	hematite	hematite (featureless spectra)			malachite					
calcite	hematite	hematite (featureless spectra)								
muscovite	hematite	hematite (featureless spectra)			muscovite					
kaolinite	hematite	hematite (featureless spectra)						kaolinite		
chlorite	hematite	hematite (featureless spectra)								
biotite	hematite	hematite (featureless spectra)								

Table 3. Spectral dominance in intimate mixture of complex mineral associations

Complex mixture or associations											
Mineral 1	Mineral 2	Mineral 3	Mineral 4	Mineral 5	Mineral 6	Mass ratios of minerals					
						1	1	1	1	1	1
hematite	chrysocolla	muscovite	–	–	–	hematite (featureless spectra)					
hematite	chrysocolla	kaolinite	–	–	–	kaolinite					
hematite	malachite	calcite	–	–	–	malachite					
muscovite	biotite	chlorite	–	–	–	muscovite					
hematite	malachite	kaolinite	–	–	–	malachite/kaolinite					
malachite	muscovite	hematite	–	–	–	malachite/muscovite					
chlorite	biotite	calcite	–	–	–	chlorite					
calcite	hematite	muscovite	–	–	–	muscovite/calcite					
chrysocolla	biotite	hematite	–	–	–	hematite (featureless spectra)					
biotite	hematite	chlorite	–	–	–	hematite (featureless spectra)					
biotite	muscovite	calcite	–	–	–	muscovite/calcite					
kaolinite	calcite	hematite	–	–	–	kaolinite					
muscovite	hematite	kaolinite	–	–	–	muscovite/kaolinite					
chlorite	biotite	calcite	hematite	–	–	chlorite/calcite					
chlorite	biotite	calcite	muscovite	–	–	muscovite/calcite					
kaolinite	calcite	hematite	muscovite	–	–	muscovite/kaolinite					
hematite	biotite	muscovite	chlorite	calcite	–	hematite (featureless spectra)					
malachite	calcite	hematite	muscovite	kaolinite	chrysocolla	hematite (featureless spectra)					

Implication to ore sorting and strategy development

The main objective of this research is to understand whether individual minerals can be identified when they occur together and are scanned within the same spectral range, and if their preconcentration is feasible. From analysis of spectra of various mixtures, the following observations were made: hematite drowns out chlorite, biotite and calcite features for all realistic proportions of these minerals. Hence, where absorption feature of any of these minerals is visible, the spectra will indicate almost complete absence of hematite. Chrysocolla is only visible in hematite at 90% concentration relative to hematite. At equal concentration, malachite and muscovite dominate hematite by showing their best diagnostic absorption feature near 2275 nm for malachite and 2200 nm for muscovite.

Both chrysocolla and malachite, which are the copper-bearing NIR-active minerals, can be targeted together by selecting the best diagnostic absorption feature of chrysocolla near 2270 nm. This is true since the feature is common at any given ratio

of their mixtures. Chrysocolla is more NIR-active than calcite, while muscovite spectral dominance over chrysocolla and vice versa is concentration dependent. Kaolinite is more readily accessible to NIR radiation than chrysocolla across all mixing ranges, with spectra appearing mixed at higher chrysocolla concentration. Chrysocolla, chlorite, hematite and/or biotite are better preconcentrated together. While chrysocolla and chlorite show absorption features side-by-side in a spectrum, chrysocolla in concentration with biotite display featureless spectra across the mixing range. This is similar to spectra of chlorite and biotite mixtures.

Malachite dominates both chlorite and calcite features at any given ratio. Malachite is relatively more NIR-active than hematite, as it shows one of its features at relatively higher hematite concentration (from ratio 4:6, malachite-hematite), while malachite features appear mixed with kaolinite. For malachite and biotite, at higher biotite concentration (90%), spectra appear featureless. Malachite dominates muscovite at higher concentration relative to muscovite, while their spectra appear mixed at higher muscovite concentration.

The spectral analysis of calcite mixed with malachite, chrysocolla, chlorite or hematite suggests that identification of calcite in such mixtures is only feasible for freely occurring calcite. Calcite dominates biotite across the mixing range. At higher calcite concentration, calcite shows features side-by-side those of kaolinite and muscovite but calcite is completely masked in the spectra from equal ratios. Hence, all three minerals can be preconcentrated together by either selecting 2200 nm feature to discriminate high -OH -low calcite or 2340 nm to discriminate high calcite -low -OH.

Though kaolinite is differentiated from muscovite by its double absorption features as against muscovite single feature (Hunt, 1979; Hunt and Hall, 1981), complete discrimination between both minerals cannot be achieved. This is true since both minerals share the same wavelength position (2200 nm) as their best diagnostic absorption feature position (Iyakwari et al., 2013). The visibility of the double kaolinite features in the mixtures with muscovite is concentration dependent. Both muscovite and kaolinite dominate chlorite and biotite.

Chlorite, apart from showing features side-by-side with chrysocolla and dominating calcite, is weak in mixtures with other NIR-active minerals. The NIR spectra of mixtures of chlorite and biotite are featureless and hence display a combined sensitivity. Where mineralogy is unknown, the spectra can be misinterpreted to represent a strongly absorbing NIR-active mineral without features. This is also true for the chrysocolla-biotite mixtures. Therefore, except in the biotite mixtures with chrysocolla and chlorite, biotite does not show its absorption features or any spectral influence in any mixture. Hence, of all minerals and mixtures investigated in this work, the spectral analysis indicates that biotite is the weakest of all the NIR-active minerals. According to Clark (1995), the iron content in biotite is responsible for masking the -OH absorption feature at shorter wavelengths near 1400 nm. Therefore, the weakness or absence of biotite features in the spectra of biotite mixed with other iron bearing minerals is ascribed to increasing iron concentration.

In general, calcite features are also invisible in the complex mixtures involving three or more minerals, where hematite, malachite or chrysocolla are present in concentration. The mixtures of strongly absorbing hematite and strongly reflecting muscovite or kaolinite indicate that at equal proportions or ratios the highly reflective hydroxyl minerals show their best diagnostic absorption feature near 2200 nm. The spectral analysis also confirms strong hematite absorption at longer NIR wavelengths (1300 to 2500 nm), reducing the overall level of reflectance and possibly masking or drowning features of other NIR-active feature displaying minerals (Bishop and Dummel, 1996; Iyakwari and Glass, 2014).

Copper-bearing minerals preconcentration

For preconcentration of copper-bearing minerals such as malachite and chrysocolla, the fact that spectra appear mixed and in some instance featureless implies that only freely occurring calcite, muscovite and/or kaolinite spectrum can be targeted for removal/reduction as a waste. Therefore, four options based on associations of the copper-bearing minerals with or without high iron bearing minerals (non-feature displaying NIR-active minerals) using NIR are developed in Fig. 6.

The first option (Fig. 6) considers silicate copper bearing minerals (chrysocolla) occurring without high iron-rich minerals (hematite). Because chrysocolla shows features side-by-side to those of kaolinite and muscovite when these occur in isolation of hematite, the spectrum displaying features diagnostic of chrysocolla (2270 nm), with or without kaolinite (2160 and 2200 nm) or muscovite (2200 nm) features, is considered as a product. Hence, the spectrum showing the calcite feature (2340 nm) is classified as a waste, since calcite displaying spectrum in chrysocolla particles indicates high grade calcite (approximately 100%). Where the spectrum appears showing features of calcite in addition to kaolinite or muscovite, given that chrysocolla has more features drowning ability on calcite even at higher calcite concentration, the spectrum shall be classified as the waste.

The second option (Fig. 6) considers copper carbonate-bearing minerals (malachite) when occurring without iron-rich minerals. Since malachite shows features side-by-side those of kaolinite and muscovite, NIR spectrum showing features near 2275 nm with or without 1415, 2200 and 2360 nm will be classified as the product. While the spectrum showing exclusively features near 2340 nm diagnostic of calcite are targeted as the waste. This is so because calcite (2340 nm) is completely masked or displaced by malachite. Therefore, similar to chrysocolla, only freely occurring CO_3^{2-} features near 2340 nm shall be considered as the waste, as any spectrum showing calcite features indicate absence of malachite. The third option (Fig. 6) considers both copper-bearing minerals (chrysocolla and/or malachite) occurring in association with high iron-bearing minerals (hematite). Since high iron-rich minerals with either chrysocolla or malachite may result in a featureless spectrum, featureless spectra may indicate the product for relatively high concentrations of hematite. This also applies to biotite occurring with chrysocolla and/or chlorite. Therefore, in

addition to the featureless spectra, the spectra showing features diagnostic of either or both chrysocolla or malachite near 2270 nm and 2275 nm respectively, with or without 1415, 1915 or 2360 nm, shall also be considered as the product. Hence, only spectra showing features near 2200 nm (muscovite and kaolinite) or 2160 nm (kaolinite only) and 2340 nm (calcite) are classified as the waste. This option is targeted at removing (reducing) both calcite and clay minerals. Note that the choice of only the 2340 nm feature may also target low-grade clays (kaolinite and muscovite), selecting both feature (2200 and 2340 nm) will target both low-grade and high-grade calcite and clays minerals. This option should only be chosen if high purity of minerals fractions is more important than a high recovery: low-grade but valuable minerals (malachite or chrysocolla) are likely to be locked in muscovite or kaolinite.

Finally, when spectra appear to be similar, being either featureless or showing diagnostic features of all NIR-active minerals, the ore will not be sortable using a NIR sensor.

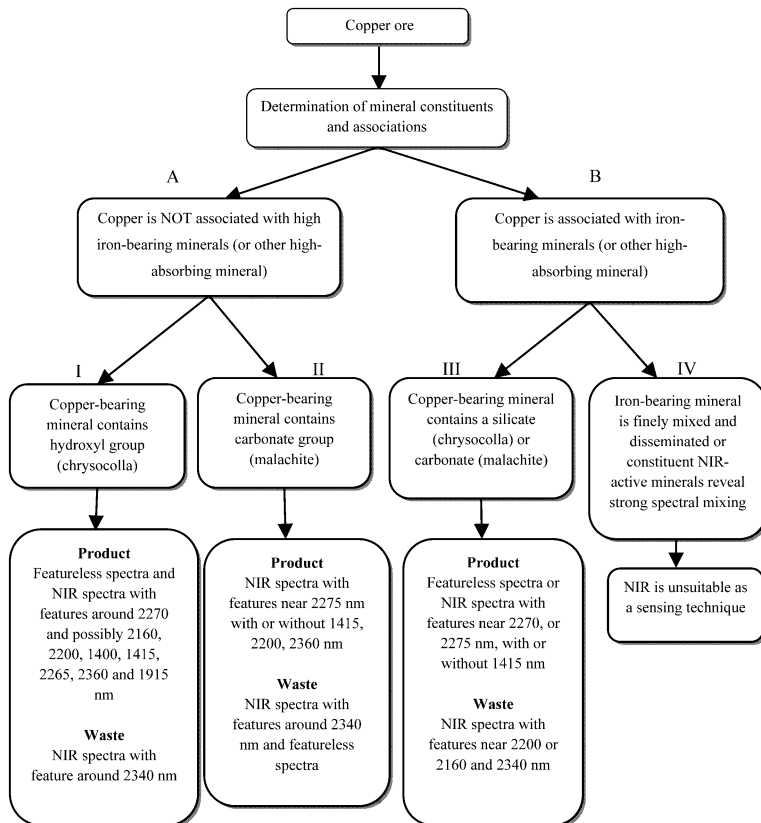


Fig. 6. Framework for NIR sorting for copper ore, based on the characteristics of an individual spectrum (modified from Iyakwari et al., 2013)

Hematite preconcentration

For preconcentration of hematite from clay (e.g. muscovite, kaolinite) and/or carbonate (calcite) minerals, the absence of absorption features (2200 nm) will indicate the dominance and purity of hematite particle. Hence, preconcentration can be achieved by upgrading hematite and reducing water consumption during washing. Though the absence of 2200 nm absorption features is indicative of product (hematite) and the presence of the absorption features indicative of presence of clay minerals (waste), the waste fraction of this preconcentration strategy shall require washing instead of disposal. Discarding either muscovite or kaolinite bearing mineral may lead to valuables sent to waste, when the clay absorption feature (2200 nm) is present in spectra at equal hematite-muscovite or hematite-kaolinite concentration.

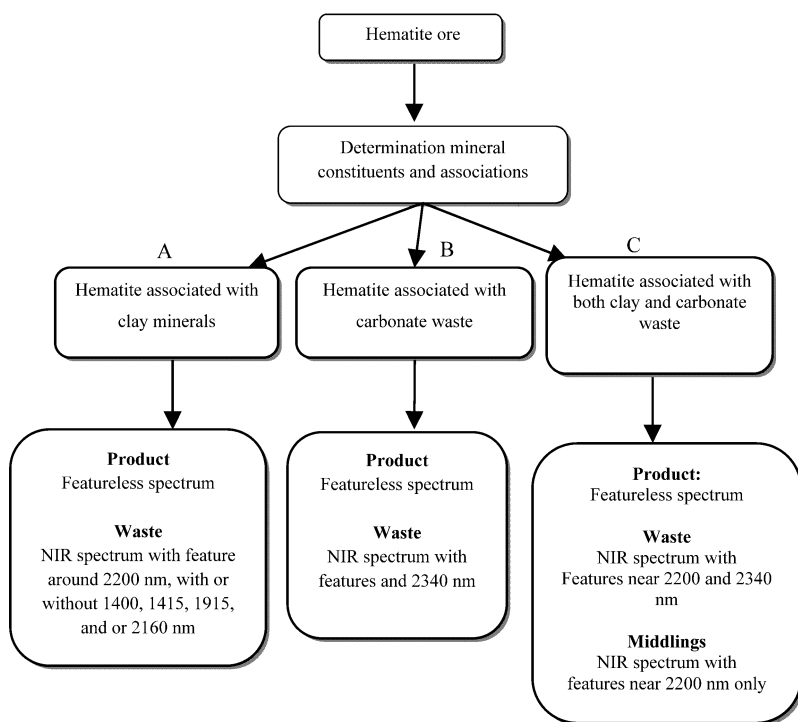


Fig. 7. Framework for NIR sorting for hematite ore using characteristics of individual spectra

In the case of carbonate (calcite) removal, particles displaying calcite feature (2340 nm) shall be classified as waste not requiring further treatment, since calcite does not show features in the presence of hematite. It should also be noted that spectra not displaying calcite features does not mean the absence of calcite. Therefore, while preconcentration of hematite from calcite may give high recovery, the grade of ore is likely to be low due to dilution. Hence, the product fraction will require further upgrading. A strategy is presented in Fig. 7.

For the determination of moisture content in kaolinitic clays containing either hematite minerals or other NIR-active minerals without absorption features, the presence of hematite is likely to mask the water features (1915 or 1840 nm and/or 1415 nm), creating the impression that clay samples are moisture free (dry). As an indirect way of determining the moisture content in kaolinitic clay sample, the presence of hematite or any high spectral absorbing mineral in concentration could also indicate the presence of moisture, as the absence of water features does not necessarily imply a dry sample.

Conclusion

The strategies outlined depend upon the ore type, constituent NIR-active mineralogy and character, as such may need to be either calibrated or modified for specific ore type to achieve optimal results. Therefore, in order to scope an application, a good understanding of constituent minerals, minerals associations and diagnostic features locations of the NIR-active minerals in the ore is essential. The aim of using NIR as a preconcentration method is to upgrade an ore by either eliminating or reducing the quantity of overall waste material reporting to the next processing stage. Using the NIR, this can only be achieved by reducing the waste fraction, since most minerals are dominated by others. In this investigation carbonate (calcite) and/or clay (muscovite/kaolinite) rich particles are the only groups of waste minerals that can be targeted by NIR, since other non-copper-bearing minerals like chlorite, biotite and hematite cannot be effectively discriminated from chrysocolla or malachite. The same is true for hematite ores, as only clay and carbonate minerals can be reduced from a feed.

The absence of water (or moisture) features in the NIR spectra does not imply that the moisture content of kaolinite bearing sample is zero. Hence, NIR is not recommended for direct moisture content determination in clays known to contain either hematite or other NIR-active mineral without diagnostic absorption features.

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