3 Description of Minerals and Milling Techniques

3.1 Minerals

Specimens of microcline originating from Port Hedland, Western Australia supplied by Commercial Minerals (currently trading as Unimin), Perth, WA, biotite from Bancroft, Canada and hornblende from Kragero, Norway supplied by Wards Scientific, Rochester, NY, USA were used in this study. These are common rock forming minerals and contain elements that may be made available to plants by dissolution of the minerals in soil solution. They are also of differing mineral structures and thus provide a range of silicate mineralogy for this study. The chemical composition of each mineral was determined by XRF and the structural formulae of the minerals used are presented in *Table 3.1*. Although the composition of the microcline indicates it is alkali feldspar, it will be termed a microcline throughout this thesis.

Table 3.1: Structural formula of minerals used in this study.

Mineral	Structural Formula
Port Hedland Microcline	$(Na_{0.29}K_{0.65})(Al_{0.99}Si_{3.01})O_8$
Bancroft Biotite	$(K_{1.90}Na_{0.16})(Mg_{3.22}Fe_{2.40}Mn_{0.12})(Al_{1.95}Si_{6.15})O_{20}(OH,F)_4$
Kragero Hornblende	$(K_{0.11}Na_{0.71})Ca_{1.73}(Mg_{3.27}Fe_{1.55}Mn_{0.01})(Al_{1.35}Si_{7.01})O_{22}(OH)_2$

3.2 Attrition Milling Techniques

Wet milling was chosen for this research in order to reduce agglomeration of particles (Papirer and Roland, 1981) and the ability to produce greater surface area when compared to dry milling, although a greater degree of amorphization is achieved in the latter (Juhász and Opoczky, 1990).

Three phases of milling were undertaken during milling based on the volume of material to be milled:

- SPEX* milling of a small volume of mineral for characterisation and the dissolution experiment (Chapters 4 and 5);

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* SPEX is a trademark of Syntech Coproration, Houston TX

- Small Attritor Mill for larger volumes to be used in the plant growth experiment (Chapter 6); and
- A large attritor mill for evaluating milling costs (Chapter 7).

Although all three milling techniques involved wet milling, the ratio of water to mineral, the composition and ratios of the milling media, and the type of agitation varied depending on the mill characteristics. Differences between these milling techniques are discussed below and milling techniques are compared in Chapter 7.

3.2.1 Mineral Preparation

Impurities were removed from crushed mineral specimens prior to milling. Each mineral was initially ground in a steel Tema ring mill for 1 minute and passed through a 90µm sieve. This was considered the starting material (sample at 0 minutes attrition milling).

3.2.2 SPEX Mill

A SPEX 8000 shaker mill was initially used to evaluate the effect of milling on mineral properties and to prepare minerals for the dissolution experiment. Shaker mills are appropriate for attrition milling of small samples and use high frequencies and small amplitudes of vibration (Fecht, 1996). A 80cm^3 hardened steel mortar was loaded with 50g of 5mm hardened steel balls of density 10 g cm^{-3}, 5g of mineral and 15ml of MilliQ water, and shaken for times of 1, 6 and 24 hours. After milling, the minerals were washed into 50ml centrifuge tubes with MilliQ water and centrifuged at 6000rpm for 20 minutes. The supernatant was collected and the mineral placed in an oven at 60ºC to dry. The supernatant was analysed for K (microcline and biotite), Mg (biotite and hornblende) and Ca (hornblende) by atomic absorption spectrometry (AAS) to determine the amount of mineral dissolved during milling, assuming that no precipitation or sorption of dissolved ions had taken place.

3.2.3 Small Attritor Mill

Larger volumes of mineral were milled in a vertical stirred ball mill (attritor). The mill is constructed from 316 stainless steel with a diameter of 70mm and depth of 46mm, producing an effective mill volume of 165mL. A 20mm diameter central shaft constructed from 253MA stainless steel with arms constructed from high-speed tool steel stirred the grinding media. The arms have a diameter of 8 mm, with a spacing of 20 mm, arranged in a vertical spiral with 90º between consecutive blades. The diameter of the stirring arm was 60 mm. The mill containing 185g of 4.8 mm hardened steel balls of density 10g cm^{-3}, 30g of mineral and 90mL of deionised water was attached to a drill press and stirred at 1490 rpm for periods of 1, 6 and 24 hours. Samples were washed with deionised water into 50ml centrifuge tubes, and centrifuged at 11,000 rpm for 5 minutes. The supernatant was removed and the sample dried at 60ºC.

3.2.4 Large Attritor Mill

One kilogram of the microcline feldspar was milled in a larger attritor mill to evaluate milling costs. The mineral was supplied from Commercial Minerals already ground and passed through a 75µm sieve. The mill is constructed from 316 stainless steel with a diameter of 155mm and depth of 400mm, producing an effective mill volume of 7L. A 40mm diameter central shaft constructed from 253MA stainless steel with arms constructed from high-speed tool steel stirred the grinding media. The arms have a diameter of 12 mm, with a spacing of 24mm, arranged in a vertical spiral with 45º between consecutive blades. The diameter of the stirring arm was 126 mm. The mill containing 12kg of 6mm alumina balls of density 3.5g cm^{-3}, 1kg of mineral and 2L of deionised water was stirred at 500 rpm for 6 hours. Samples were collected at periods of 15 and 30 minutes and 1, 2, 4 and 6 hours and dried at 60ºC.

3.3 Sample Designations

A summary of designations of milled minerals used throughout this thesis is presented in *Table 3.2*.

Table 3.2: Sample designation of milled minerals used in this study.

	Initial Mineral	1 hour milling	6 hour milling	24 hour milling
Microcline	M0	M1	M6	M24
Biotite	B0	B1	B6	B24
Hornblende	H0	H1	H6	H24