Australia's National
Science Agency

XRD analyses of MinQ Zero samples for crystalline silica content, March to June 2024

Aaron Seeber, David McCallum & Winston Liew
CSIRO Report No: EP2024-3347
4 September 2024

Allan Zhang, Talostone

Commercial-in-confidence

Introduction

Twenty-one samples were submitted for XRD analyses, with reference to crystalline silica content, as summarised in Table 1.

Table 1: Sample Numbers

Client Reference	Original Report Number	Original Sample Number
Arabescato Borghini 9500	2024-2888	248645-12
Arabescato Nero 9600	2024-2888	248645-02
Arctic White 3010	2024-2888	248645-01
Calacatta Gold 9080	2024-2534	248634-2
Calacatta Luxe 9030	2024-2534	248634-1
Calacatta Oro 9040	2024-1256	248583-1
Calacatta Statuario 9050	2024-1545	248597-3
Carrara Classic 8110	2024-1545	248597-7
Carrara Gioia 8080	2024-2534	248634-3
Carrara Gold 8150	2024-1545	248597-8
Concrete Original 9010	2024-2534	248634-4
Desert White 6010	2024-2888	248645-05
Elba White 8300	2024-2888	248645-06
Imperial Danby 9020	2024-2534	248634-5
Lord White 9400	2024-2888	248645-07
Marmo Gris 8250	2024-1545	248597-9
Onyx White 9100	2024-2888	248645-09
Serene White 8060	2024-1711	248602-3
Super White 9300	2024-1545	248597-6
Taj Mahal 9060	2024-1545	248597-1
White MirrorLux 5020	2024-2888	248645-11

Sample Preparation and Analysis Conditions

The samples were ground as follows: Each sample was passed through a 6 inch jaw crusher at the widest aperture (~12 mm) and then screened over a 12# (3.36 mm) Tyler 200 mm sieve. The oversize was then returned to the jaw crusher and progressively crushed by reducing the jaw aperture over time. Once the material was all 7 mm or less, it was split using a laboratory riffler to produce a representative sample for milling (~100 g). That representative sample was milled in a 200 mm zirconia mill on a Rocklabs pulveriser for ~ 2 minutes. Each representative sample was then finer than 75 microns. A ~10g aliquot was then taken and micronized for 20 minutes under ethanol in a McCrone micronizing mill, after which the slurry was dried at 80°C. Each resultant dried aliquot was then loaded into a standard specimen holder prior to the collection of the X-ray diffractogram.

A Bruker D8 Advance A25 X-ray Diffractometer operating under either CoKα or CuKα radiation (40kV, 40mA) equipped with a Lynx Eye XE-T detector was employed to obtain the X-ray diffractograms. For the Cu instrument, the aliquots were scanned over the 2θ range 5° to 130° with a step size of 0.02 at and a count time of 1.6 seconds per step while being spun at 15 RPM during data collection. For the Co instrument, the aliquots were scanned over the 2θ range 5° to 130° with a step size of 0.02°, variable divergence slits programmed to illuminate 8mm of the sample surface, and a count time of 1 second per step while being spun at 20 RPM during data collection.

Analyses were performed on the collected XRD data using the Bruker XRD search match program EVA™7. Crystalline phases were identified using the ICDD-JCPDS powder diffraction database. Rietveld analyses were performed on the data using the Bruker TOPAS™ V7 program to determine wt% values. Background signal was described using a combination of Chebyshev polynomial linear interpolation function and 1/x function, and signal from amorphous content was modelled using a series of pseudo-Voight functions. Cell parameters, vertical sample displacement, peak full width at half maximum, preferred orientation and scale factor were all refined. For wt% values, error ranges were calculated on the basis of three estimated standard deviations as calculated by TOPAS, for which the data was compared with that obtained from a well crystallised corundum external standard for amorphous content determination.

Results

Phases identified among the diffractograms collected from these samples are summarised in Table 2. Phase quantification results for these samples are shown in Table 3, along with wt% values. Where duplicate analyses were performed, the highest total crystalline silica content has been reported. The samples were all dominated by amorphous content, with trace to minor quantities of quartz, cristobalite and rutile also observed amongst these samples and minor gibbsite in two samples. Although no quartz could be seen in the diffractograms collected from the gibbsite-containing samples, the major quartz diffraction peak coincides with a minor gibbsite peak and as such the presence of up to 0.1wt% quartz may be masked by the presence of gibbsite. No tridymite was observable in any of the samples within the sensitivity limit of the technique.

Conclusion

Crystalline phase identification using XRD has been performed for samples obtained from the provided materials. The bulk of each sample was found to be amorphous, with the total crystalline silica content in each listed in Table 3. These test results are only reflective of the provided material and are only applicable to the specific batches from which they were obtained, on the assumption that Talostone have provided a representative sample from each batch.

Table 2: Phases found in the diffractograms collected from these samples.

Pattern #	Compound Name	Nominal Formula	Lattice	Space Group
PDF 04-005-4718	Quartz	SiO_2	Hexagonal	P3121 (152)
PDF 04-007-4907	Cristobalite	SiO_2	Tetragonal	P41212 (92)
PDF 04-005-5815	Rutile	TiO_2	Tetragonal	P42/mnm (136)
PDF 04-016-3819	Gibbsite	$Al(OH)_3$	Monoclinic	P21/n (14)

Table 3: Phase quantification results in wt% by sample.

Client Reference	Quartz	Cristobalite	Rutile	Gibbsite	Amorphous	Total Crystalline Silica
Arabescato Borghini 9500			0.9±0.1		99.1±0.1	<0.1
Arabescato Nero 9600			0.7±0.1		99.3±0.1	<0.1
Arctic White 3010	0.11±0.05		2.2±0.1		97.7±0.1	0.11±0.05
Calacatta Gold 9080	0.4±0.1		0.2±0.2		99.4±0.2	0.4±0.1
Calacatta Luxe 9030	0.7±0.1		0.4±0.2		98.9±0.3	0.7±0.1
Calacatta Oro 9040	0.36±0.07		0.4±0.1		99.2±0.1	0.36±0.07
Calacatta Statuario 9050	0.20±0.05		0.14±0.09		99.7±0.1	0.20±0.05
Carrara Classic 8110	0.18±0.06		0.3±0.1		99.5±0.1	0.18±0.06
Carrara Gioia 8080	0.28±0.09				99.72±0.09	0.28±0.9
Carrara Gold 8150	0.20±0.08		0.4±0.1		99.3±0.1	0.20±0.08
Concrete Original 9010	0.4±0.2		0.7±0.2		98.8±0.2	0.4±0.2
Desert White 6010			2.1±0.1		97.9±0.1	<0.1
Elba White 8300	0.33±0.05		0.45±0.09		99.2±0.1	0.33±0.05
Imperial Danby 9020	0.5±0.2		0.4±0.3		98.9±0.3	0.7±0.1
Lord White 9400			3.7±0.1		96.3±0.1	<0.1
Marmo Gris 8250	0.16±0.05		0.05±0.05		99.8±0.1	0.16±0.05
Onyx White 9100			1.3±0.1		98.7±0.1	<0.1
Serene White 8060			0.75±0.08	12.2±0.3	87.0±0.3	≤0.1
Super White 9300	0.16±0.05		0.06±0.06		99.8±0.1	0.16±0.05
Taj Mahal 9060	0.49±0.07	0.06±0.03	0.12±0.07		99.3±0.1	0.55±0.10
White MirrorLux 5020			0.14±0.03	2.8±0.1	97.0±0.1	≤0.1

As Australia's national science agency and innovation catalyst, CSIRO is solving the greatest challenges through innovative science and technology.

CSIRO. Unlocking a better future for everyone.

Contact us

1300 363 400
+61 3 9545 2176
csiroenquiries@csiro.au
www.csiro.au

For further information

CSIRO Manufacturing
Aaron Seeber
+61 3 9545 2958
aaron.seeber@csiro.au
www.csiro.au/manufacturing