Effects of Gibbsite, Kaolinite and Al-rich Goethite as Alumina Sources on Silico-Ferrite of Calcium and Aluminium (SFCA) and SFCA-I Iron Ore Sinter Bonding Phase Formation

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The relative effects of gibbsite, kaolinite and aluminous goethite as alumina sources on the thermal stability, concentrations and formation mechanisms of silico-ferrite of calcium and aluminium (SFCA and SFCA-I) iron ore sinter bonding phases, was investigated using in situ X-ray diffraction. Iron ore containing gibbsite as the primary source of alumina is less likely to form high quality sinter due to the lower reactivity of the alumina leading to low amounts of SFCA-I and SFCA bonding phases being generated. Sintering of this ore is likely to require higher fuel as higher temperatures are required to generate the bonding phases. Alumina in the form of kaolinite or aluminous goethite, however, produced larger amounts of both SFCA-I and SFCA and at lower temperatures. Use of kaolinite resulted in the formation of a highly reactive gehlenite intermediate phase that maximised the formation of SFCA-I, the matrix phase that imparts high strength and good reducibility characteristics to sinter. Iron ore containing aluminous goethite also generated SFCA bonding phases however the difference in the reaction mechanism between kaolinite and aluminous goethite containing ore led to less SFCA-I being formed overall. These findings give some insight into why sintering investigations using Australian ores with kaolinite tend to show less impact on sinter quality than the more widely reported alumina studies involving gibbsite-rich ores.

KEY WORDS: iron ore sinter; SFCA; SFCA-I; alumina; gibbsite; kaolinite; aluminous goethite; in situ X-ray diffraction; phase formation mechanisms.

1. Introduction

Iron ore sinter constitutes the major proportion of blast furnace ferrous burden in most countries in the Asia-Pacific region. Low temperature fluxed iron ore sinter mainly contains unreacted hematite nuclei (around 30 vol%) in a matrix (about 70 vol%) consisting of primary and secondary hematite, secondary magnetite, silico-ferrite of calcium and aluminium (SFCA), silicates and glass. Studies on SFCA morphology have further identified two different types of SFCA, i.e., SFCA and SFCA-I.1–3) SFCA-I typically appears platy, needle-like or fibrous in cross-section, whereas SFCA tends to take a columnar or blocky form. Previous work has demonstrated that SFCA-I is the more desirable bonding phase in iron ore sinter resulting in higher sinter strength and better reducibility.2,4–6)

The quality of iron ore sinter is dictated by the structure formed during the sintering process, as well as the nature, abundance and mutual interactions of the mineral phases present in the sinter.5–7) As a result, considerable importance has been placed on the chemical composition and consistency of iron ore fines, particularly the alumina content which can dissolve in all major mineral phases present in sinter except the unreacted hematite.8) Furthermore, alumina stabilises the SFCA matrix phases with SFCA-I formation favoured when the total alumina and silicate contents are low and the hematite content is high. Conversely, SFCA formation is favoured by lower iron and higher alumina and silica contents.5)

Alumina-containing iron ores are typically divided into two types, namely ores with alumina mainly as gibbsite (Al(OH)₃) and ores with alumina mainly in the form of aluminosilicates such as kaolinite (Al₂Si₂O₅(OH)₄).9) Indian ores5,10–12) and African ores13) are typically gibbsite-rich. In comparison, Australian iron ores are more often kaolinite-rich but may contain significant amounts of alumina associated with goethite.14) Extensive work on the effects of alumina on sinter structure, quality and productivity has shown that too much alumina impairs sinter quality, leading to low strength and poor low temperature reduction degradation characteristics.5,7,15,16) However, closer examination of this work shows that most studies have focused on gibbsite-rich ores or chemical reagents.5,7,11,17,18) Only limited work has used ores containing alumina in the form of kaolinite5,19,20) or aluminous goethite20) and these found a less deleterious effect on sinter quality than has been reported for ores containing gibbsite. It seems reasonable to postulate that iron
ores containing alumina in different mineralogical forms will behave differently during the sintering process, leading to differences in sinter structure and quality. In particular, given the known effects of alumina on stabilising SFCA and SFCA-I, it may be expected that different alumina types will have a significant impact on the formation and stability of these key matrix bonding phases.

A number of recent investigations have utilised in situ powder X-ray diffraction (XRD) on synthetic sinter mixtures to establish the fundamental formation mechanisms of SFCA and SFCA-I. In each of these studies gibbsite was used as the alumina source. As indicated, however, iron ores may also contain significant alumina in the form of kaolinite and alumino goethite. The aim of the current investigation is to clarify the role of different types of alumina present in iron ore fines. This was achieved by replacing gibbsite in synthetic iron ore fines mixtures with alternative alumina sources in order to compare their reactivity and effects on the formation and stability of SFCA and SFCA-I phases under simulated sintering conditions.

2. Experimental

2.1. Sample Preparation

The gibbsite and kaolinite mixtures were prepared from synthetic hematite Fe₂O₃ (Acros Organics, 99.999%), calcite CaCO₃ (Thermo Fisher, 99.95%), quartz SiO₂ (Sigma Aldrich, 99.995%), gibbsite Al(OH)₃ (Alcan OP25 Super White, 99.9%) and kaolinite Al₂Si₂O₅(OH)₄ (Aldrich, 96.4%). The phase homogeneity of each of the starting materials was confirmed by XRD before preparation of the mixtures. Each of the weighed powders was mixed under ethanol in a McCrone micronising mill (McCrone Research Associates, London, approximately 2 min g⁻¹) in order to achieve particle sizes (~10 μm) suitable for XRD-based quantitative phase analysis (QPA). Mixtures were centrifuged, the excess ethanol decanted and the residue dried overnight in an oven at 333 K. Table 1 shows the bulk compositions of the gibbsite and kaolinite mixtures, determined by X-ray fluorescence spectroscopy (XRF).

A synthetic aluminous goethite material was prepared using procedures detailed in Schwertmann and Cornell. The resulting material was washed in 0.4 M HCl (50 ml of acid to 2.5 g sample) on a reciprocating shaker (70–80 rpm) for 30 mins. The slurry was then centrifuged, the acid decanted and the residue was washed with deionised water. The resulting material was washed in 0.4 M HCl (50 ml of acid to 2.5 g sample) on a reciprocating shaker (70–80 rpm) for 30 mins. The slurry was then centrifuged, the acid decanted and the residue was washed with deionised water.

The solid/water slurry was then dialysed in deionised in water for 5 days according to the method of Cornell and Schneider. The Al concentration in the purified aluminous goethite powder was ~3 atom%, and as a result it was not possible to match the composition of the aluminous goethite mixture with the preceding gibbsite and kaolinite mixtures. An additional kaolinite mixture (kaolinite-mix2) was therefore prepared with a composition comparable to the aluminous goethite mixture (Table 1) to allow comparisons to be made.

2.2. In situ XRD Experimentation and Quantitative Phase Analysis

In situ XRD experiments were performed using an INEL diffractometer, which incorporates a CPS120 position sensitive detector allowing for simultaneous collection of up to 120° 2θ of diffraction data. The Cu tube was operated at 40 kV and 35 mA. An Anton Paar HTK10 high-temperature chamber employing a Pt resistance strip heater containing a sample well measuring ~20 × 7 × 0.2 mm was fitted to the diffractometer. Samples were heated over the range 293–623 K under a flow of a 0.5 vol% O₂ in N₂ gas mixture (to give a nominal oxygen partial pressure of pO2 = 5 × 10⁻³ atm). The oxygen partial pressure was chosen based on the work of Hsieh and Whitehead, who determined that this pO2 maximised the formation of Ca-rich ferrites whilst still producing mineral assemblages similar to those found in industrial sinters. XRD data were collected over the range 10° ≤ 2θ ≤ 120° continuously throughout heating, with individual datasets collected for 1 min.

A heating rate of 16 K min⁻¹ was used from 293–873 K. The rate was then reduced to 8 K min⁻¹ for the 873–1023 K interval, after which a rate of 4 K min⁻¹ was used for the interval 1023–1623 K which corresponded to the predominant period of Ca-rich ferrite phase formation and decomposition. The temperatures of phase formation/transformation given throughout the manuscript are those at the start – which is when the temperatures were automatically recorded – of the relevant data set. The uncertainty in these temperature values, therefore, was the difference between the temperatures at the start of successive data sets. The decomposition of precursor phases and the formation of new phases as the experiments progressed was visualised by stacking the datasets to produce plots of accumulated data with temperature plotted vs 2θ, viewed down the intensity axis.

Rietveld refinement-based QPA was performed on the individual datasets using TOPAS (Version 5). The crystal structure data of were used for Fe₂O₃, CaCO₃, Al(OH)₃, α-SiO₂, Al₂Si₂O₅(OH)₄, FeOOH, β-SiO₂, CaO, C₂F, CaAl₂SiO₄, CF, CₐF, γ-CaF₂, SFCA-I, SFCA and Fe₂O₃, respectively. The use of the QPA algorithm embodied in TOPAS returns relative, rather than absolute, concentrations for crystalline phases in a system if amorphous material, including melt phases, are present. Previous work has demonstrated that amorphous Al₂O₃ is present in these systems after the decomposition of Al(OH)₃. Therefore, absolute phase concentrations as a function of temperature were determined, via an ‘external standard’ methodology.

| Table 1. XRF-determined bulk compositions, in mass% oxides, of the synthetic mixtures. |
|-----------------|---------|---------|---------|---------|
| Mixture         | Fe₂O₃  | CaO     | SiO₂    | Al₂O₃   |
| Gibbsite        | 76.80  | 15.38   | 4.84    | 2.78    |
| Kaolinite-mix1  | 76.62  | 15.27   | 4.98    | 2.77    |
| Aluminous       | 78.42  | 16.05   | 5.16    | 1.11    |
| Goethite        | 76.80  | 15.38   | 4.84    | 2.78    |
| Kaolinite-mix2  | 77.99  | 16.17   | 4.53    | 1.13    |
3. Results and Discussion

3.1. Phase Changes during Heating

Figure 1(a) shows a plot of accumulated in situ XRD data, viewed down the intensity axis and with temperature plotted vs $2\theta$, for the experiment performed using the gibbsite mixture. The first event was the decomposition of Al(OH)$_3$ to amorphous Al-oxide which was completed by 569 K. Then, the transformation of $\alpha$-SiO$_2$ to $\beta$-SiO$_2$ occurred. This was completed by 861 K which is within experimental uncertainty of the 846 K reported by Kihara$^{47}$ and gives confidence in the accuracy of the temperature measurement. CaCO$_3$ then decomposed to CaO, which was completed by 915 K. The first Ca-rich ferrite to form was dicalcium ferrite at ~1 073 K (i.e. 2CaO·Fe$_2$O$_3$, which is abbreviated hereafter as C$_2$F). This was followed by CF (i.e. CaO·Fe$_2$O$_3$) and a ternary CFA phase (composition determined by Webster et al.,$^{23}$) at ~1 243 K. As the temperature increased further, $\gamma$-CFF (nominally Ca$_3$Al$_2$SiO$_7$), SFCA-I and SFCA all formed, at ~1 373 K, 1 398 K, and 1 433 K, respectively. The $\gamma$CFF phase had disappeared by ~1 460 K while SFCA-I persisted until ~1 473 K. The stability range of SFCA extended to 1 503 K, and thereafter melting produced a phase assemblage of Fe$_3$O$_4$ in a CaO–SiO$_2$–Al$_2$O$_3$-rich melt.

Figure 1(b) shows the plot of accumulated in situ XRD data for the kaolinite-mix1 mixture. The decomposition of the crystalline Al$_2$Si$_2$O$_5$(OH)$_4$ was completed by ~823 K. The significant points of difference between the phase behaviour observed in the kaolinite-mix1 experiment, compared to the gibbsite experiment, are: i) the formation of gehlenite (Ca$_2$Al$_2$SiO$_7$) at ~1 173 K; ii) the lack of formation of CF, and $\gamma$CFF; and iii) the significant increase in the thermal stability range of SFCA-I, with this phase first observed at ~1 323 K and observed up until ~1 498 K. The thermal stability range of SFCA was similar in the gibbsite and kaolinite-mix1 experiments, with SFCA first observed at ~1 423 K, and with melting of the SFCA complete by 1 503 K.

Figure 1(c) shows the plot of accumulated in situ XRD data for the aluminous goethite mixture. The decomposition of the Al-FeOOH phase was complete by ~623 K. Gualtieri and Venturelli$^{48}$ reported that the decomposition of goethite (FeOOH) resulted in the formation of protohematite, an intermediate phase which is Fe-deficient. Protohematite retains residual hydroxyl ions for charge balance and gradually transforms to Fe$_3$O$_4$. In this current study no attempt has been made to separate contributions from Fe$_3$O$_4$ and protohematite to the ‘hematite’ reflections in the in situ XRD data. The first Ca-rich ferrite to form was C$_2$F at ~1 023 K followed by both CF and the ternary CFA phase at ~1 200 K. As the temperature increased further, $\alpha$-SiO$_2$ formed at ~1 293 K and observed up until ~1 323 K. The stability range of SFCA was similar in the gibbsite and kaolinite-mix1 experiments, with SFCA first observed at ~1 423 K, and with melting of the SFCA complete by 1 503 K.

3.2. Quantitative Phase Analysis (QPA)

Figure 2 shows the results of the QPA for the gibbsite mixture. It should be noted that, due to significant peak overlaps, the CFA concentration has been determined up to 1 363 K only (i.e. up until the onset of $\gamma$CFF formation). In addition, poor particle statistics precludes reliable Fe$_3$O$_4$ quantification after melting of SFCA due to a relatively small number of Fe$_3$O$_4$ crystallites in a large amount of melt resulting in inaccurate relative peak intensities of the Fe$_3$O$_4$ phase. A similar, although not so severe, particle statistical effect is also observed for the $\alpha$- and $\beta$-SiO$_2$ phases.

Fig. 1. In situ XRD data collected for the (a) gibbsite, (b) kaolinite-mix1 and (c) aluminous goethite mixtures, over the range 298–1 623 K at $p_{O_2} = 5 \times 10^{-3}$ atm. (Online version in color.)
Fe$_3$O$_4$ unit cell volume and a shift in the peaks to lower $2\theta$ values. A similar effect has been reported before \cite{23} and was rationalised on the basis that when Fe$_3$O$_4$ forms through melting of SFCA it is initially Ca-rich, but as the temperature increases the smaller Fe$^{2+}$ cation substitutes for the larger Ca$^{2+}$ cation in the Fe$_3$O$_4$ unit cell resulting in a decrease in the unit cell volume. For the gibbsite experiment, such a shift to higher $2\theta$ is not observed and so the Fe$_3$O$_4$ when it first forms is likely to have a smaller Ca content.

Figure 4 shows the results of the QPA for the aluminous goethite experiment (4a,b), and for the kaolinite-mix2 experiment (4c). The most obvious differences between these two experiments is in the abundance of SFCA-I generated with 52 mass% in the kaolinite-mix2 compared to 38 mass% in the aluminous goethite mixture. Based on the results of Webster et al. \cite{26}, the slightly higher basicity of kaolinite-mix2 (B = 3.0) compared to the aluminous goethite mixture (B = 3.1) is considered unlikely to explain the higher SFCA-I concentration attained in the experiment for the kaolinite-mix2 compared to the aluminous goethite. The higher SFCA-I concentration observed is, therefore, considered to be due to the use of kaolinite rather than aluminous goethite.

Figure 3 shows the results of the QPA for the kaolinite-mix1 mixture. The amount of SFCA-I which formed (60 mass%) was significantly greater than for the gibbsite mixture (18 mass%), whereas the amount of SFCA generated was similar (32–35 mass%). The thermal stability range of the SFCA-I was also significantly greater in the kaolinite experiment (1 323–1 498 K for the kaolinite mix1 experiment compared to 1 398–1 473 K for the gibbsite experiment). In addition, the mechanism of Fe$_3$O$_4$ formation appears different across the two experiments. For the gibbsite mixture, it appears that Fe$_3$O$_4$ forms at least in part by reduction of the Fe$_2$O$_3$ at quite low temperatures during the generation of SFCA (above ~1 423 K). In contrast, in the kaolinite-mix1 experiment Fe$_3$O$_4$ forms only during melting of the SFCA phases (above ~1 483 K). The differences in Fe$_3$O$_4$ behaviour may be due to differences in the amount of SFCA-I, and hence the amount of Al$_2$O$_3$ which is locked up in the SFCA-I until higher temperatures (i.e. the effect of alumina stabilisation). In the gibbsite experiment, the amount of unreacted Al$_2$O$_3$ is presumably greater at ~1 423 K and the ‘free’ Al$_2$O$_3$ is then available to react with Fe$_2$O$_3$ to form Fe$_3$O$_4$ at lower temperatures.

Another observation supporting the assignment of different Fe$_3$O$_4$ formation mechanisms is that for the kaolinite-mix1 experiment, after melting of SFCA is complete there is a general shift of the Fe$_2$O$_3$ peaks to higher $2\theta$ angle (see Fig. 1(b)). A shift in the opposite direction is expected during heating alone, with heating causing an increase in the Fe$_3$O$_4$ unit cell volume and a shift in the peaks to lower $2\theta$ values. A similar effect has been reported before \cite{23} and was rationalised on the basis that when Fe$_3$O$_4$ forms through melting of SFCA it is initially Ca-rich, but as the temperature increases the smaller Fe$^{2+}$ cation substitutes for the larger Ca$^{2+}$ cation in the Fe$_3$O$_4$ unit cell resulting in a decrease in the unit cell volume. For the gibbsite experiment, such a shift to higher $2\theta$ is not observed and so the Fe$_3$O$_4$ when it first forms is likely to have a smaller Ca content.

Another feature of the SFCA-I concentration curve in Fig.
4(c) for kaolinite-mix2 is that C$_2$F appears to be involved in the reaction to form SFCA-I, whereas in (4b) the C$_2$F phase is completely consumed before SFCA-I begins to form. C$_2$F was also observed to be involved in the reaction to form SFCA-I in Fig. 3 for the original kaolinite-mix1 composition. The formation of more CF in the kaolinite-mix2 experiment compared with the original kaolinite-mixture (i.e. a decreasing Al$_2$O$_3$ concentration results in a reduction in both SFCA-I and SFCA contents).

3.3. Implications for Sintering of Alumina-containing Iron Ores

Mineralogical results reported in the literature using different types of alumina, including chemical reagents, kaolinite, and gibbsite as the alumina sources agree on the essential role of alumina in the formation of sinter structure, particularly SFCA.$^{6,21}$ However, while there is widespread agreement from mineralogical studies of high alumina sinters, the effect of alumina on sinter quality varies, depending on the type of alumina present in iron ores. Previous studies on the effect of alumina in the form of gibbsite on sinter quality demonstrate that sinter strength deteriorates as the alumina content increases even though the alumina promotes the formation of SFCA which should be beneficial to sinter strength.$^{49–51}$ Lower strength of the mineral constituents in the sinter due to the presence of high alumina content in their lattices has been quoted as the main cause responsible for the lower sinter strength observed with gibbsite.$^{59}$ Furthermore, un-reacted gibbsite powders in the sinter may form centres of weakness and result in weaker sinter.$^{59}$ In comparison, from the limited sinter quality data available from iron ores containing kaolinite as the main source of alumina, it appears that alumina in the form of kaolinite is less detrimental to sinter strength and reduction degradation (RDI) compared to gibbsite-type alumina.$^{18–20}$

The current investigation involving an analysis of the matrix phases formed during sintering when using iron ores containing different alumina sources, sheds significant light on these observed differences in the quality of the sinters produced. These are summarised as follows:

1. Gibbsite-rich ores – the reactivity of alumina sourced from gibbsite is low. Alumina is initially taken up by the formation of CFA and partly by Fe$_3$O$_4$ which forms and is stabilised at a lower temperature (almost simultaneously with SFCA and SFCA-I) in the gibbsite experiment. The lack of availability of alumina for participation in SFCA and SFCA-I forming reactions results in smaller amounts of these phases being generated. Iron ores containing alumina primarily in the form of gibbsite would therefore be expected to generate sinters of low quality due to the low abundance of total SFCA phases. Also, the results suggest that higher sintering temperatures are required to begin forming SFCA and SFCA-I in gibbsite-rich iron ores and, once formed, there is a smaller window of optimum operating temperature before the two phases begin to decompose to magnetite and melt. The thermal tolerance for maximising SFCA-I and SFCA bond phase production is limited to around 100–120 K (i.e. between ~1 398 and 1 503 K) before melting occurs;

2. Kaolinite-rich ores – the reactivity of alumina sourced from kaolinite is comparatively high with the reaction kinetics favourable to the production of large amounts of SFCA-I and SFCA. The formation of both phases is enhanced by the initial reaction of the alumina- and silica-rich kaolinite with lime to produce gehlenite. This phase appears to be highly reactive toward hematite leading to the formation of SFCA-I at temperatures as low as 1 323 K and SFCA
at 1423 K. The alumina in kaolinite has more of an effect on the generation of SFCA-I compared to SFCA, which, from a sinter quality point of view, is advantageous given the recognised higher strength and reduction degradation characteristics in sinters characterised by high SFCA-I contents.\(^1,6\) A comparison of results from the kaolinite-mix1 and kaolinite-mix2 compositions shows that less gehlenite was formed in the kaolinite-mix2 due to the lower overall alumina content. This also translated into less SFCA-I and SFCA being formed; and

3. Aluminous goethite-rich ores – the reactivity of alumina sourced from aluminous goethite behaves most like that of the alumina from kaolinite. A key difference however is that less SFCA-I is formed when aluminous goethite is the source (~38 mass%) compared to kaolinite (~52 mass% for mix2). Furthermore, SFCA-I formation was less rapid in the aluminous goethite mixture compared to that observed at high temperatures in the kaolinite mixture. It is likely that these differences are due to the different SFCA-I formation mechanisms as noted previously.

On the basis of the test results, iron ores in which the primary source of alumina is gibbsite (e.g. Indian and African iron ores) are less likely to form high quality sinter due to the low reactivity of the alumina leading to low amounts of SFCA-I and SFCA (for a given alumina blend level). Furthermore, these ores are likely to require a greater heat input as higher temperatures are required to generate the bonding phases. These findings align with previous observations regarding the influence of gibbsite by De et al.\(^{49}\) and Das et al.\(^{50}\) who reported that significantly higher fuel rates were required to maintain reasonable sinter quality. This, however, will significantly increase coke breeze consumption leading to environmental and economic costs. In contrast, ores containing alumina in the form of kaolinite or aluminous goethite (e.g. Australian iron ores) have been shown to promote the formation of SFCA and SFCA-I bonding phases at lower temperatures and with extended thermal stability before melting. These findings provide some insight into why the limited pilot-scale sintering testwork with ores containing kaolinite and aluminous goethite showed much less effect of alumina on sinter quality than has been reported for ores with gibbsite.\(^{19,20}\)

In the QPA experiments the extension of the thermal stability range of SFCA-I formation was similar for kaolinite and aluminous goethite. The synthetic aluminous goethite appeared to produce slightly less SFCA-I than kaolinite at the same alumina level, but more work is required to understand the effect of naturally occurring aluminous goethites in iron ores. Further work is also required to understand the different mechanism of FeO formation observed for gibbsite compared to kaolinite and aluminous goethite and the possible relationship with final sinter quality.

4. Conclusion

Due to the considerable increase in iron ore demand and the depletion of higher grade resources, the alumina content in iron ore fines from around the world is gradually increasing. Depending on the source of the iron ore, this increase in alumina comes from a range of mineralogical sources including gibbsite, kaolinite and aluminous goethite. Each has a different impact on the quality of iron ore sinter produced as the quality is directly influenced by the sinter structure which varies according to the nature, abundance and interactions of the alumina-containing mineral phases present.

The current investigation examined the effect of different types of alumina present in synthetic iron ore fines on the formation and stability of SFCA and SFCA-I, the two main bonding phases in iron ore sinter. Results showed that iron ore in which the primary source of alumina is gibbsite is less likely to form high quality sinter due to the low reactivity of the alumina leading to low amounts of SFCA-I and SFCA. Sintering of this ore is likely to require higher fuel as higher temperatures are required to generate the bonding phases. Iron ore containing alumina in the form of kaolinite or aluminous goethite produced SFCA bonding phases at lower temperatures than for gibbsite and with extended thermal stability. Kaolinite-containing ore generated a highly reactive gehlenite intermediate phase that resulted in maximum formation of SFCA-I, the matrix phase considered to impart structure which varies according to the nature, abundance and interactions of the alumina-containing mineral phases present. The authors wish to thank Dr Martin Wells (CSIRO Mineral Resources, Kensington) for providing the synthetic aluminous goethite sample and advising on the method for its purification.

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