

Iron ore production by using a new Gemini surfactant at 273K

Zhiqiang Huang,^{a,*} Wenyuan Li,^a Shuyi Shuai,^a Shiyong Zhang,^a Hongling Wang,^b
Rukuan Liu,^c Chen Cheng,^d Xinyang Yu,^a Guichun He,^a and Weng Fu^e

-
- b. *Guangdong Institute of Resources Comprehensive Utilization, Guangzhou, 510650, China*
c. *Hunan Academy of Forestry, Changsha, Hunan, 410004, China*
d. *School of Chemistry and Chemical Engineering, Central South University, Changsha, 410083, China*
e. *School of Chemical Engineering, The University of Queensland, St Lucia, 4072 QLD, Australia*

Table of contents

| | |
|---|----|
| 1. Experimental information | 2 |
| Micro-flotation experiments | 2 |
| Bench-scale flotation experiments | 2 |
| Contact angle measurements | 3 |
| FTIR-spectrum analysis | 3 |
| Zeta potential tests | 4 |
| Krafft point comparison | 4 |
| 2. Synthesis and characterization | 5 |
| Synthesis of BDB | 5 |
| ¹ H NMR spectra | 7 |
| ¹³ C NMR spectra | 8 |
| FTIR spectrum | 9 |
| Mass spectrum | 10 |
| 3. Material | 11 |
| Chemical composition analysis | 12 |
| XRD | 13 |

1. Experimental information

Micro-flotation experiments

The flotation machine used in this experiment was XFG-II flotation machine, which has a 35 ml of flotation cell (Jilin exploration machinery). Mineral (2 g) was placed in the flotation cell, which was then added to 30 mL water. After adding the collector to the flotation cell, stirred for 3 min, and the bubble was scraped for 4 min to obtain concentrate. After the concentrate into the oven drying, after weighing, calculation of flotation recovery. Repeat the operation several times and take the average value. The flotation flowsheet was shown in **Figure S1**.

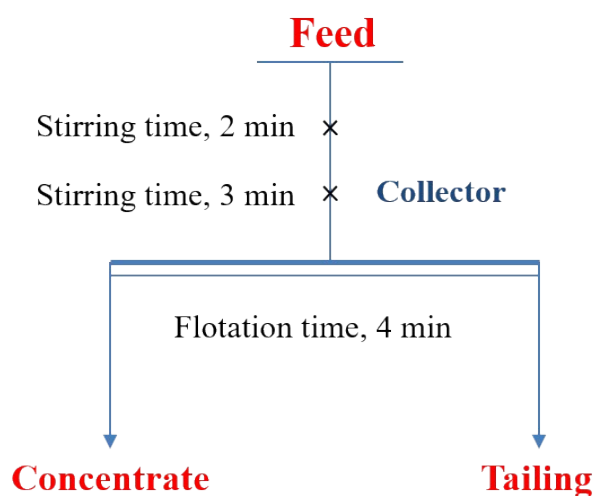


Figure S1. The flowsheet of single mine flotation experiments.

Bench-scale flotation experiments

The flotation machine selected for this bench-scale flotation was XFD mechanical flotation machine (Changchun prospecting Machinery). Magnetite (500 g) was added to the flotation cell with 800 mL tap water (298 K/273 K). After adding collector (BDB/DA), the bubbles were scraped for 4 min. The foam product was repeated twice

to obtain the final product quartz, while the tailings part was repeated once to obtain the final product magnetite. Quartz and magnetite were dried and recycled. The flotation flowsheet was shown in **Figure S2**.

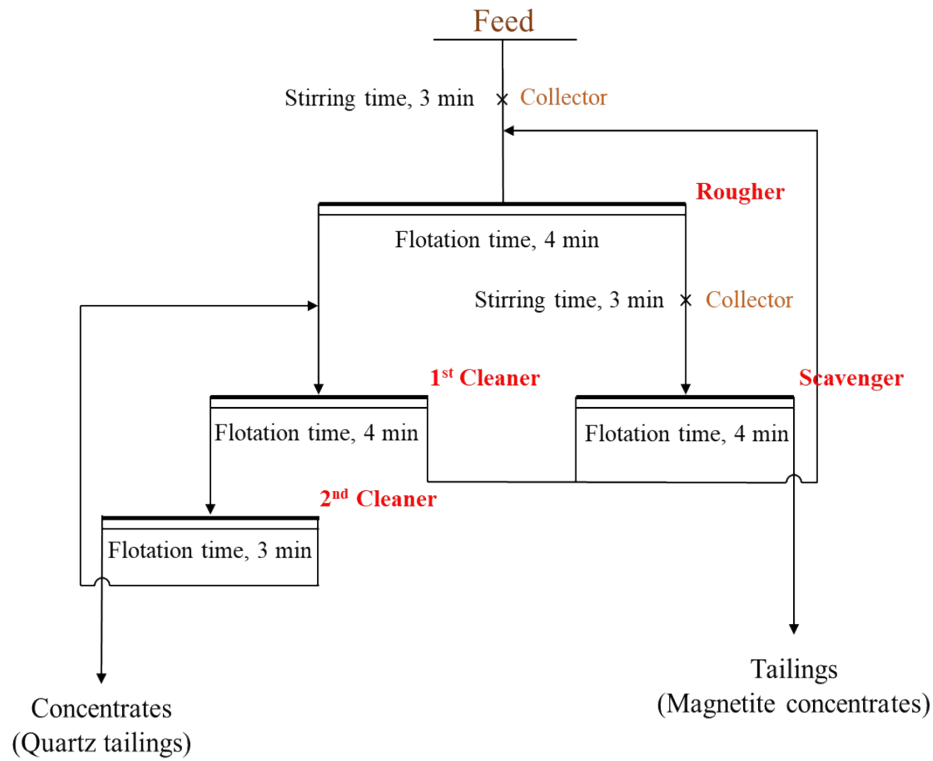


Figure S2.The flowsheet of bench-scale flotation experiments.

Contact angle measurements

The contact angle measurements using a Kruss DAS100 Shape instrument (Germany) for quartz and magnetite were performed. The change of contact angle of quartz and magnetite with BDB/DA concentration at 273 K and 298 K were measured, respectively. After polishing, quartz and magnetite were immersed in BDB/DA with adjusted concentration for 15 min. Thereafter, quartz and magnetite were taken out and dried, and the average value was taken after repeated measurement.

FTIR-spectrum analysis

2 g pure quartz and magnetite (-3000 mesh) were weighed, adding 30 mL deionized

water and stirred for 5 min. Adding BDB with a concentration of 1×10^{-4} mol/L to deionized water, adjusted pH=7, and stirred with mineral for 2 h at room temperature. Thereafter, the samples were extracted and filtered, washed with water and vacuum-dried for 24 h in an oven at 313 K. Weigh the mineral and potassium bromide at 1:100 by mass. After grinding, mixing and preparation, the absorption peaks of 500 to 4000 cm^{-1} were measured by FTIR-650 spectra.

Zeta potential tests

The Zeta potentials were tested by a JS94H Zeta potential tester (Brookhaven Instrument Corp). For each test, 2 g pure quartz and magnetite (-3000 mesh) were added to 2000 mL deionized water. After full stirring, stand for 48 h to take the upper suspension for testing. KNO_3 was added as the background electrolyte solution in all measurements. After adjusting the pH of the suspension, added BDB/DA collector and stirred for 10 min. The Zeta potential tests were carried out and the average value was taken for each pH point 5 times, with the error no more than ± 2 mV.

Krafft point comparison

Krafft point was the critical temperature for surfactant precipitation. At 298 K, dodecylamine was mixed with hydrochloric acid solution 1:1.1 and then deionized water was added to prepare 1% solution. BDB was also prepared to prepare 1% solution. Thereafter, the two were put into the cold room at 273 K to observe the crystal precipitation.

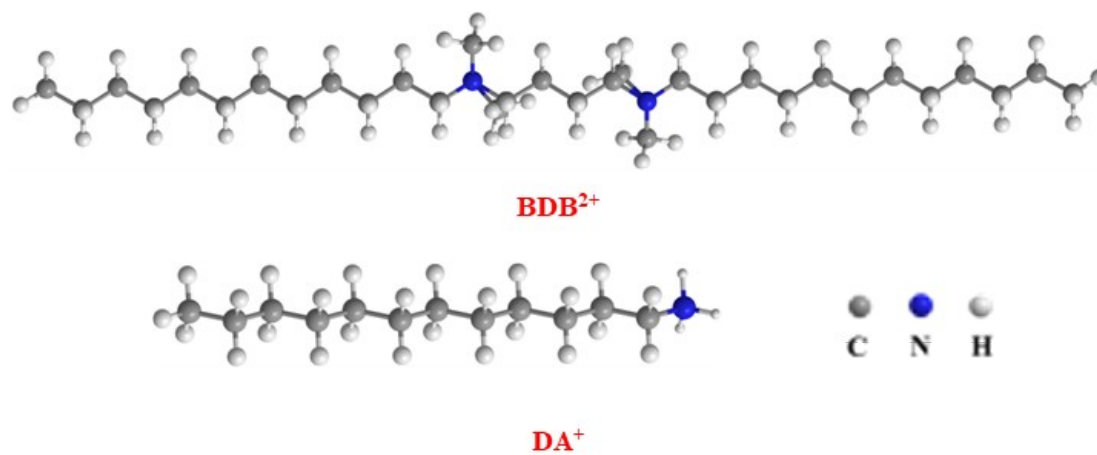


Figure S3. Synthetic route for Gemini surfactant BDB and the molecular structures of BDB²⁺ and DA⁺.

The conclusion of NMR spectrum analysis of BDB were shown in Figure 1B and Figure 1C. ^1H NMR (chloroform-d, 400 MHz, δ): 0.84 (t, 6H, 2- CH_3 , 1-H), 1.18~1.32 (m, 36H, 18 - CH_2 -, 2-H), 1.72 (m, 4H, 2 - CH_2 -, 3-H), 2.07 (m, 4H, 2 - CH_2 -, 4-H), 3.27 (s, 12H, 4 - CH_3N^+ , 5-H), 3.41 (t, 4H, 2 - CH_2N^+ , 6-H), 3.88 (t, 4H, 2 - CH_2N^+ , 7-H). ^{13}C NMR (chloroform-d, 100 MHz, δ): 14.14 (- CH_3 , a-C), 19.87 (- CH_2 -, b-C), 22.69 (- CH_2 -, o-C), 22.91 (- CH_2 -, c-C), 26.35 (- CH_2 -, d-C), 29.25 (- CH_2 -, e-C), 29.34 (- CH_2 -, f-C), 29.46 (- CH_2 -, g-C), 29.49 (- CH_2 -, h-C), 29.61 (- CH_2 -, i-C), 29.66 (- CH_2 -, j-C), 31.90 (- CH_2 -, k-C), 50.91 (- CH_3N^+ , m-C), 63.52 (- CH_2N^+ , n-C), 65.26 (- CH_2N^+ , l-C). Figure 1D was the FTIR spectrum of BDB, IR (KBr, cm^{-1}): ν 2924 (C-H), ν 2852 (C-H), ν 1471 (C-N). The result of mass spectrum of BDB was shown in Figure 1E. ESI-MS (CH_3OH): calculated for $\text{C}_{32}\text{H}_{70}\text{N}_2\text{Br}_2$ 642; found 242 [$\text{M}-2\text{Br}^-/2$], $m/z=242$ is the fragment of target compound after the removal of two bromide ions ([$\text{M}-2\text{Br}^-/2$]).

^1H NMR spectra

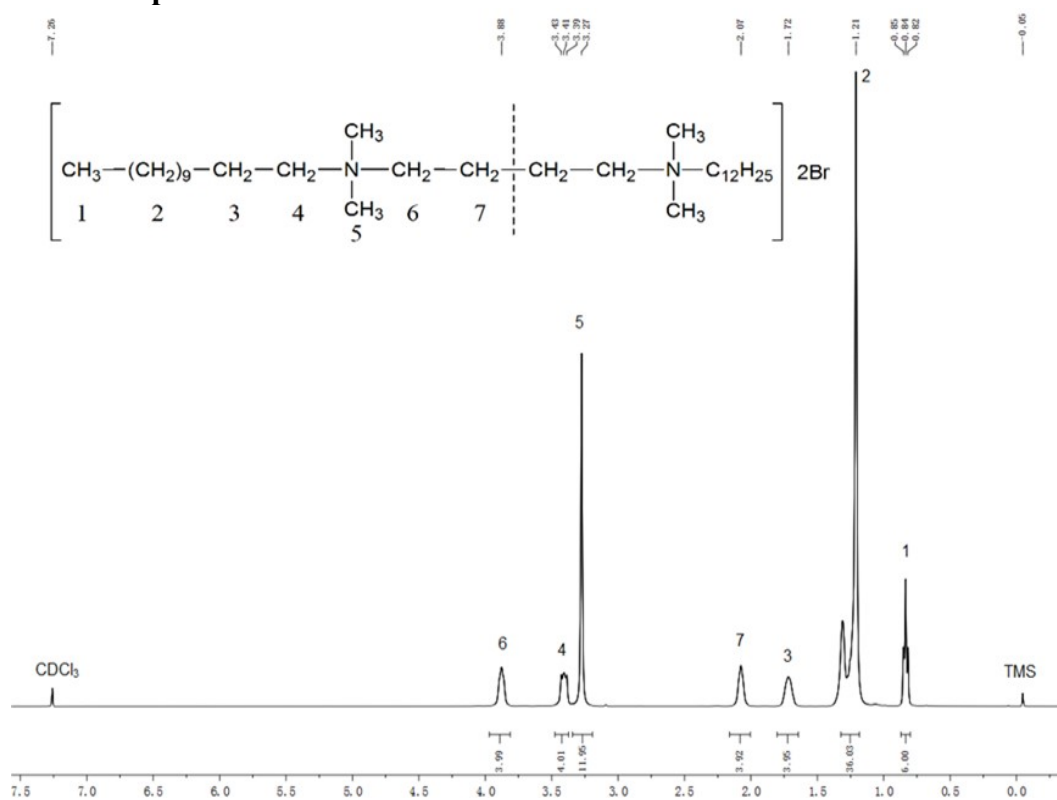


Figure S4. ^1H NMR spectra of BDB.

¹³C NMR spectra

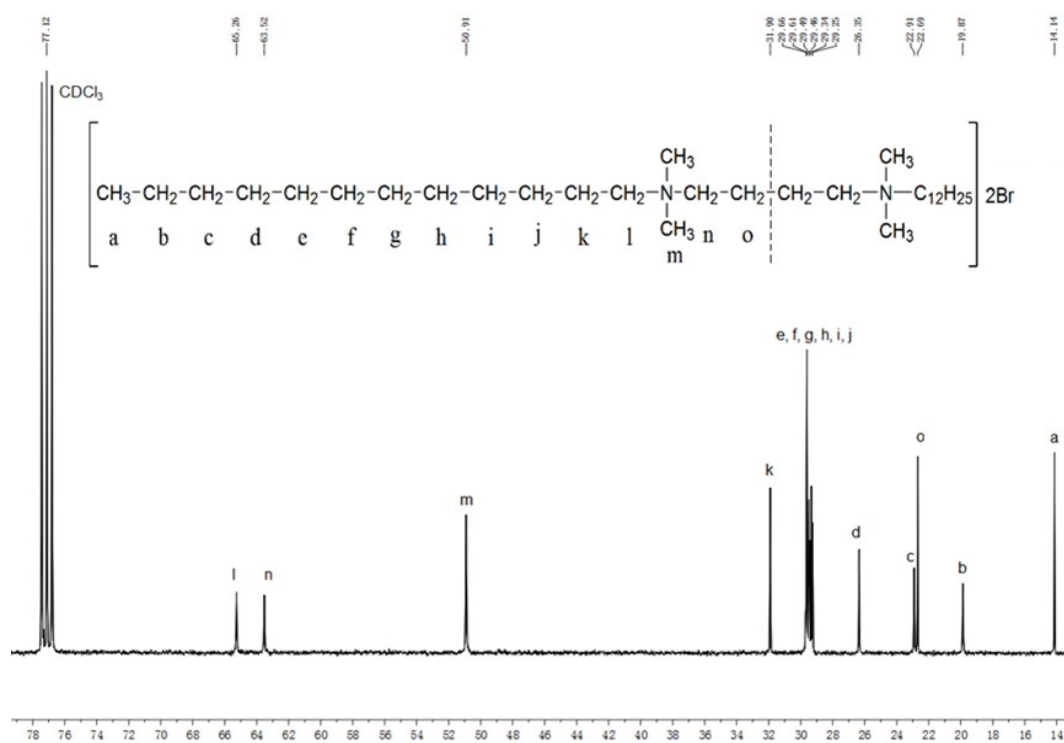


Figure S5. ¹³C NMR spectra of BDB.

FTIR spectrum

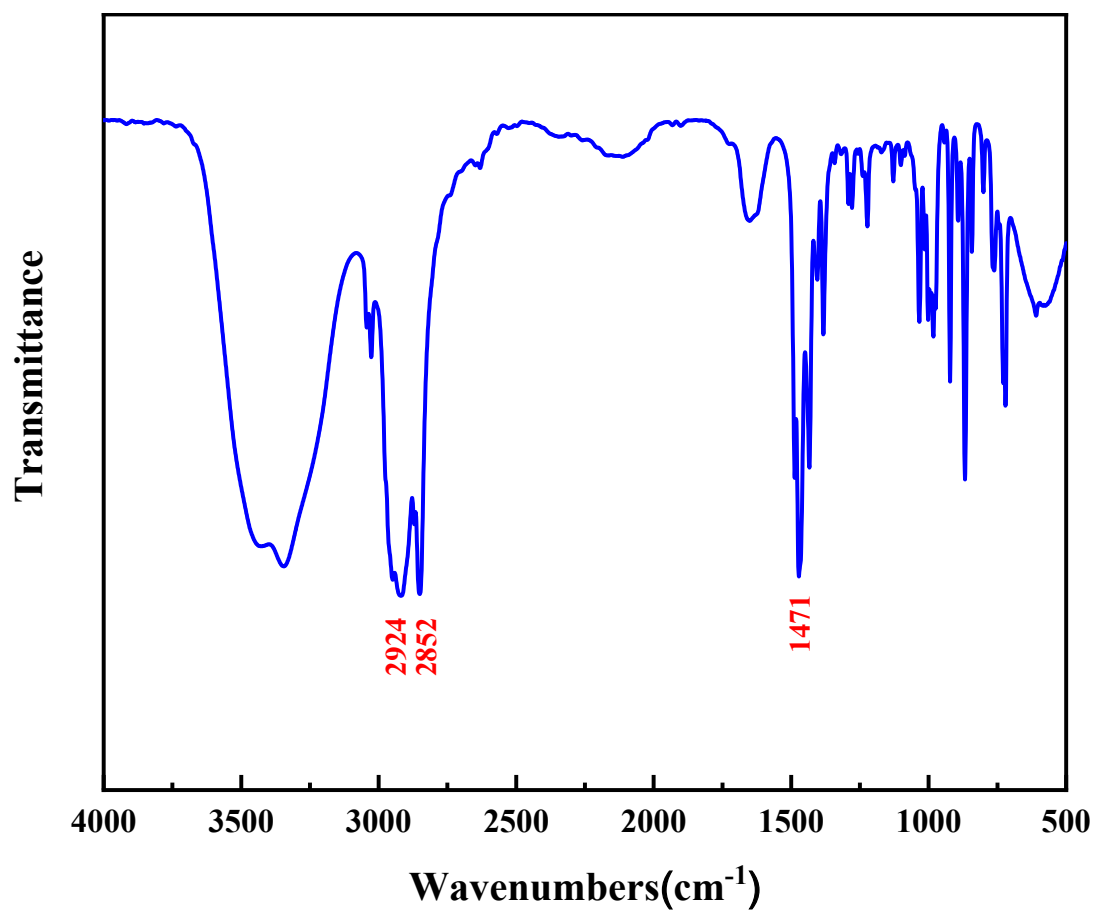


Figure S6. FTIR spectrum of BDB molecule.

Mass spectrum

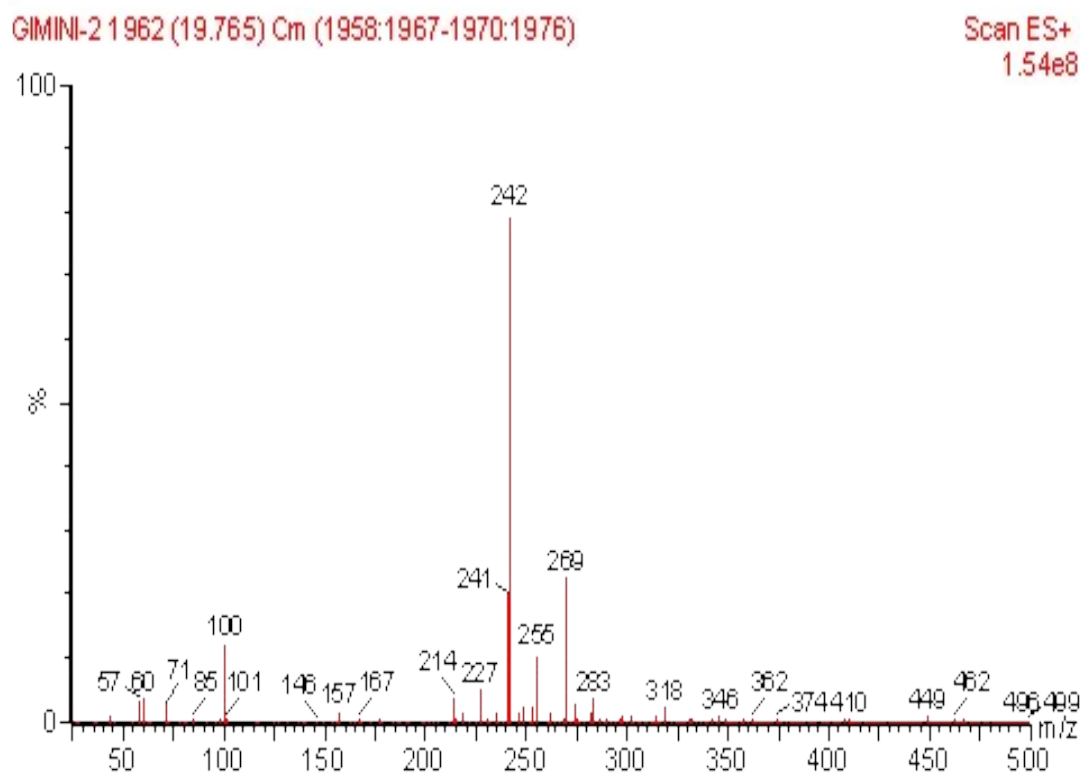


Figure S7. The result of mass spectrum.

3. Material

Pure minerals quartz and magnetite from Fujian Makeng Mining Co., LTD., China, were used in the micro-flotation test. X-ray diffraction results of quartz and magnetite were shown in **Figure S8** and **Figure S9**. Magnetite concentrate purchased at Tianye Plant, Shanxi Province (China) was used in bench-scale flotation. The mineral phase analysis results showed that the mineral contains magnetite, quartz, the content was 52.87%, 35.98%, also contains amphibole, chlorite, siderite, the content is 5.28%, 2.02%, 1.34%, in addition, there are hematite, plagioclase, potassium feldspar, dolomite, the content is 0.85%, respectively. 0.61%, 0.56%, 0.49% and the result of chemical composition analysis was displayed in **Table S1**. Minerals (-200~+400 mesh) were used for flotation experiment and XRD analysis. The water was separately frozen to 273 K~298 K to maintain a low temperature environment.

Table S1. The result of chemical composition analysis of magnetite ore.

| | | | | | | |
|-----------|-------------------|-------------------------------|-----------------|--------------------------------|------|------|
| Component | TFe | SiO ₂ | MgO | Al ₂ O ₃ | MnO | CaO |
| Content/% | 47.21 | 33.46 | 0.94 | 0.89 | 0.72 | 0.33 |
| Component | Na ₂ O | P ₂ O ₅ | SO ₃ | K ₂ O | Ig | |
| Content/% | 0.14 | 0.09 | 0.08 | 0.04 | 1.98 | |

XRD

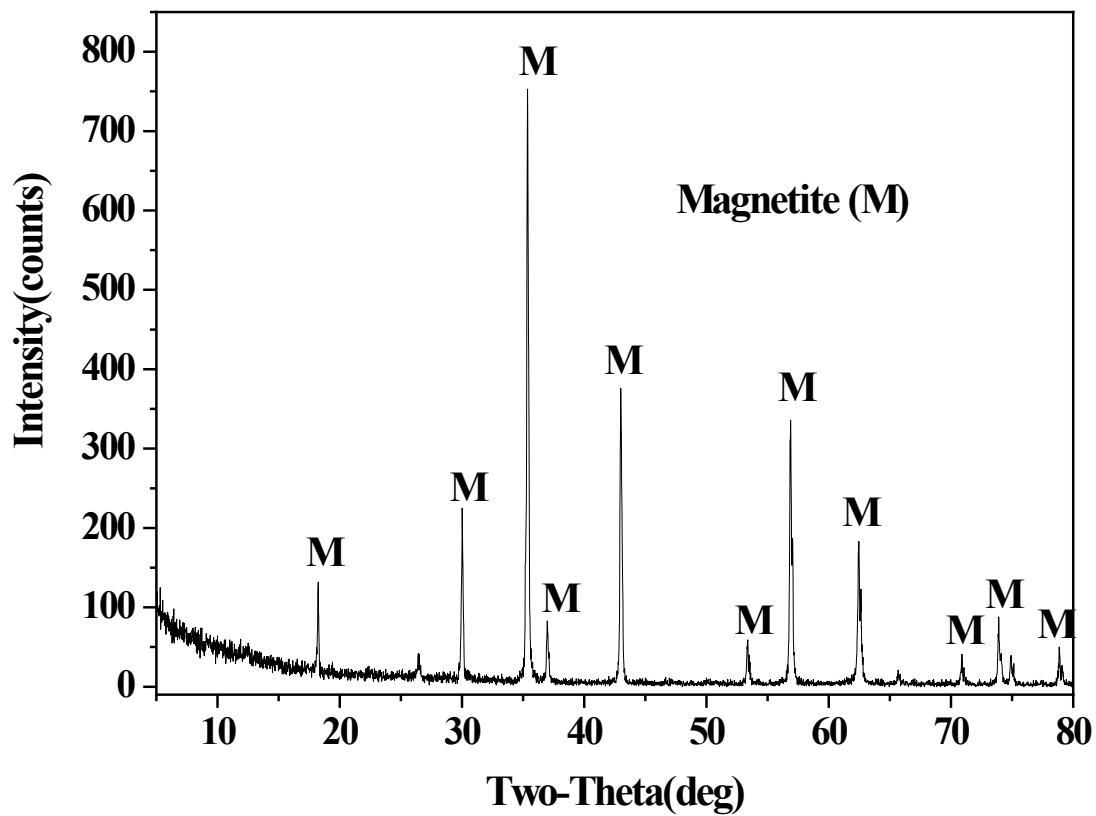


Figure S9. XRD of magnetite.