Supplementary material for

Investigation of Microstructure and Magnetic Properties of CH_4 Heat Treated Sr-Hexaferrite Powders during Re-Calcination Process

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1. Initial Sr-hexaferrite powder sample prepared by conventional method

1.1. Synthesis of initial Sr-hexaferrite powder by conventional method

Strontium carbonate was milled in a wet ball mill with alcohol medium for 20 minutes and a weight ratio of 10:1 balls to powder. Then iron oxide powder was added to the mixture with a ratio of 1:5.5 and the milling continued for 40 minutes. Then the milled mixture was kept in air to evaporate the alcohol. The obtained powder, which was a suitable mixture of iron oxide and strontium carbonate, was consequently placed in a furnace and subjected to calcination at 1100ºC for one hour with a heating rate of 10 ºC min^{-1} , and thus the initial Sr-hexaferrite powder was prepared.

1.2. Properties of initial Sr-hexaferrite powder

The X-ray diffraction pattern of the Sr-hexaferrite powder prepared by the conventional method along with the Miller indices of its crystal planes are shown in Figure S1. As can be seen, there is few traces of the α-Fe_2O_3 secondary phase.

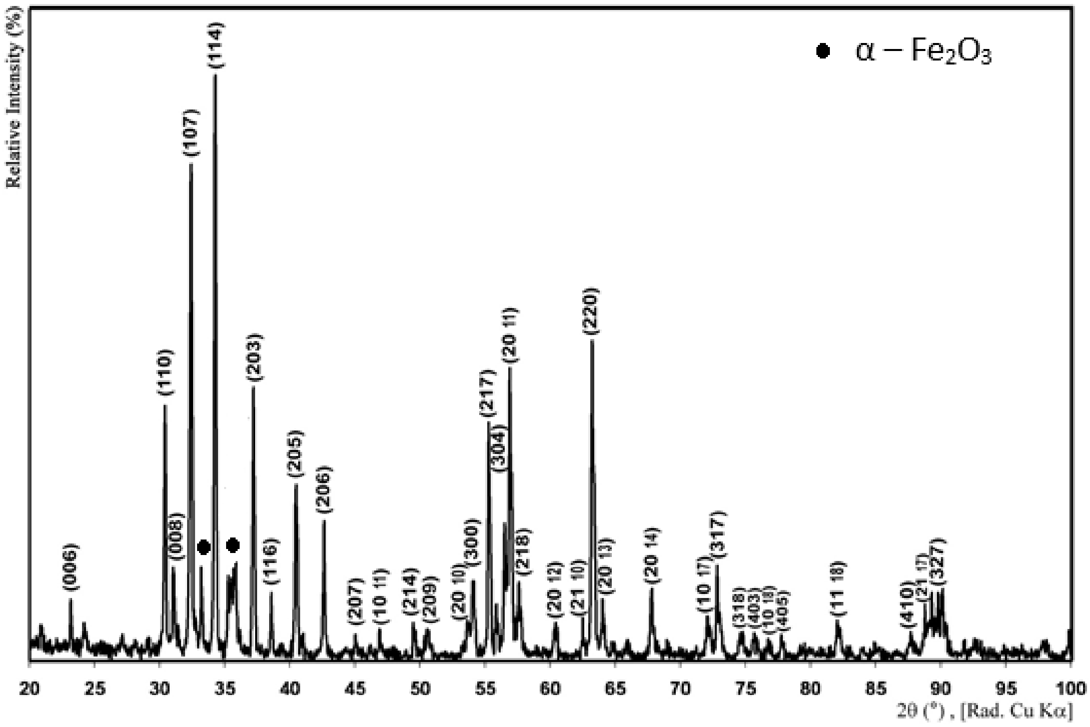

Figure S1. XRD pattern of Sr-hexaferrite powder prepared by conventional method.

Figure S2 shows the SEM image of the Sr-hexaferrite powder prepared by conventional method. As can be seen, the initial powder is composed of hexagonal-like particles.

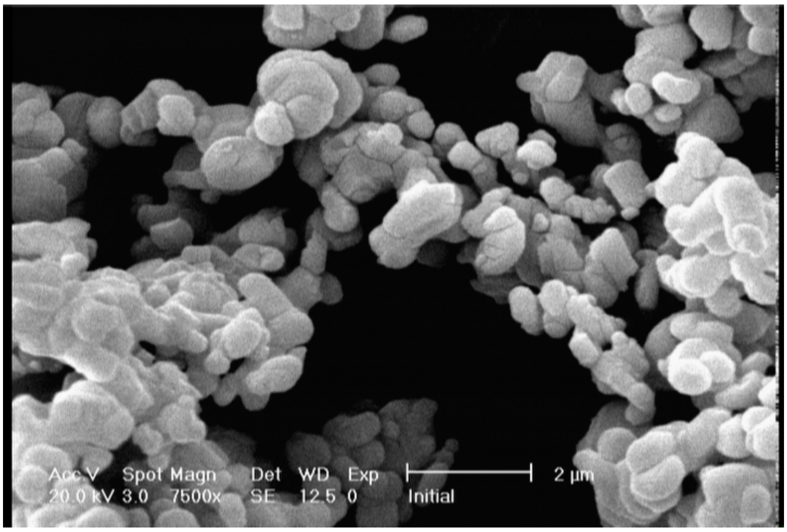

Figure S2. SEM image of Sr-hexaferrite powder prepared by conventional method.

Figure S3 shows the magnetization curve of the initial Sr-hexaferrite powder prepared by the conventional method. The magnetic properties resulting from this hysteresis curve are 60.88 emu g^{-1} saturation magnetization, 35.30 emu g^{-1} remanence and 3.5 kOe coercivity. The kink in the hysteresis loop can be due to the presence of α-Fe_2O_3 soft magnetic phase [1], which was shown in the XRD pattern (Figure S1).

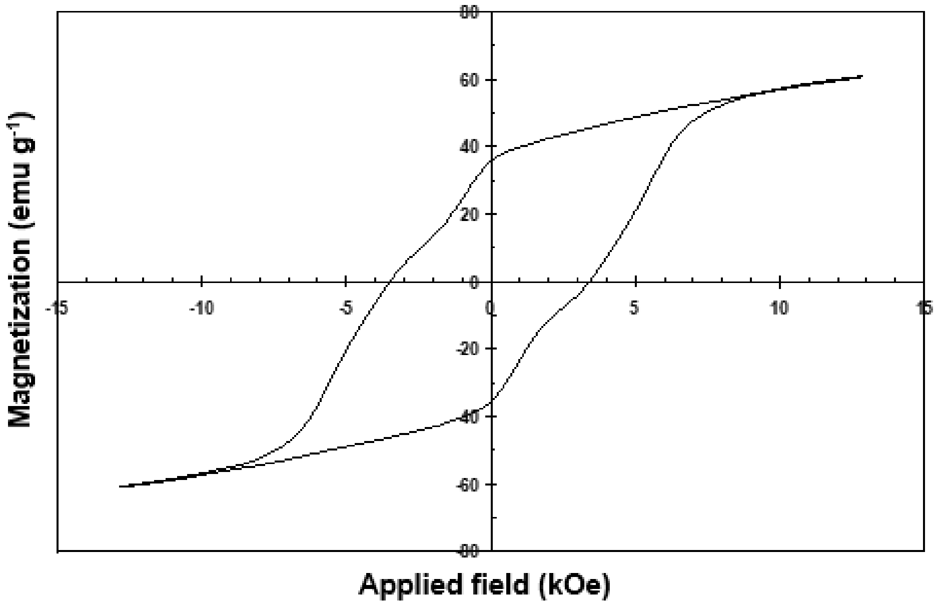

Figure S3. Hysteresis curve of the initial Sr-hexaferrite powder prepared by the conventional method.

2. The crystallite size of phases after re-calcination process using Debye-Sherrer method

Table S1. Summary of crystallite size of phases after re-calcination process with different calcination conditions; the heat treatment conditions before re-calcination for all samples were temperature: 950ºC, gas flow rate:15 cc min^{-1}, and time: 30 min.

Calcination temperature (°C)	Phase	d (nm)
200	Fe	35.54
500	$Sr_7Fe_{10}O_{22}$	30.11
800	$SrFe_{12}O_{19}$	36.62
900	$SrFe_{12}O_{19}$	32.84
1000	$SrFe_{12}O_{19}$	39.78
1100	$SrFe_{12}O_{19}$	38.33
1200	$SrFe_{12}O_{19}$	40.91

3. XRD pattern of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min)

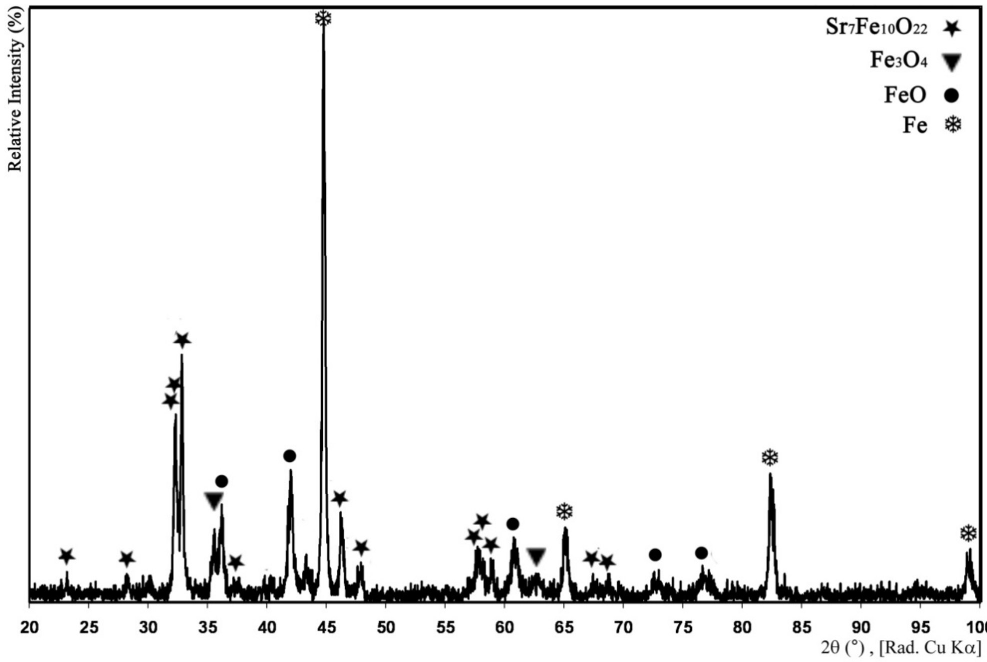

Figure S4. XRD pattern of the Sr-hexaferrite powder reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min)

4. Hysteresis curves of samples

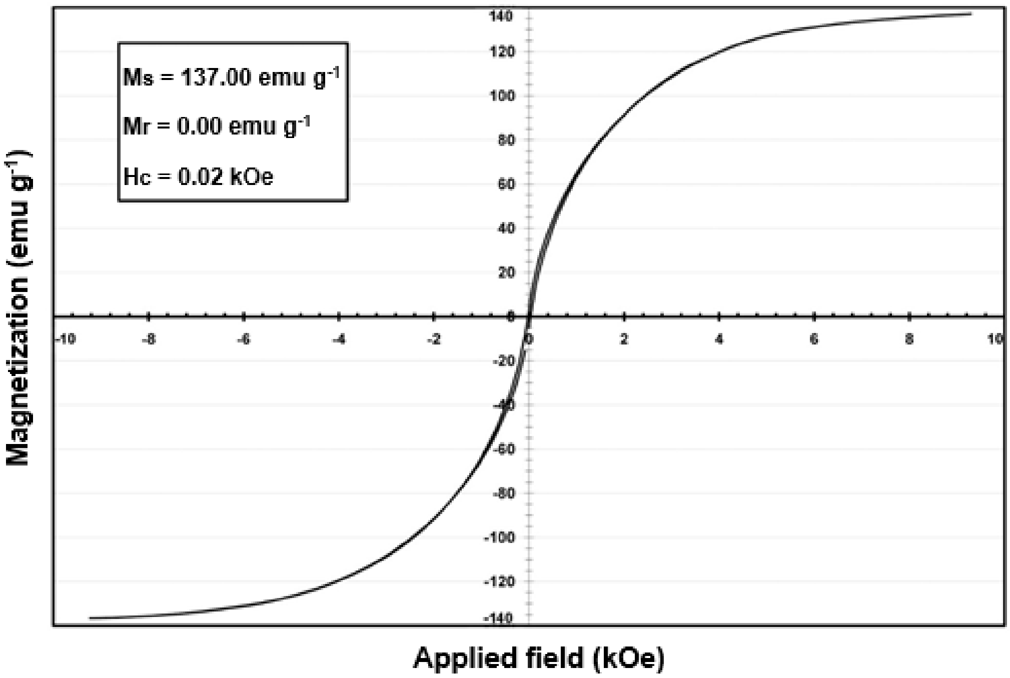

Figure S5. Hysteresis curve of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min) and then calcined at 200ºC.

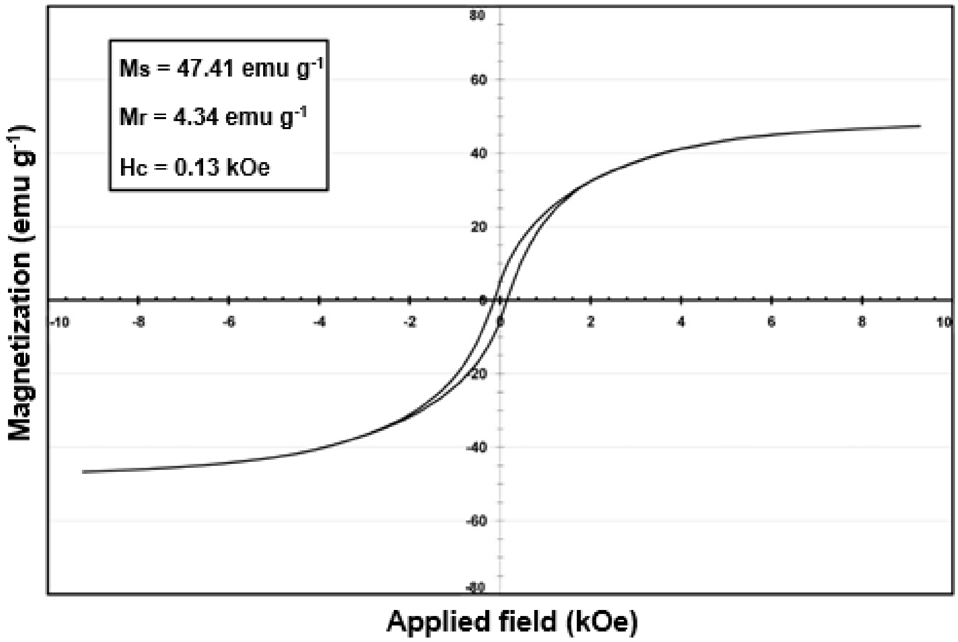

Figure S6. Hysteresis curve of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min) and then calcined at 500ºC.

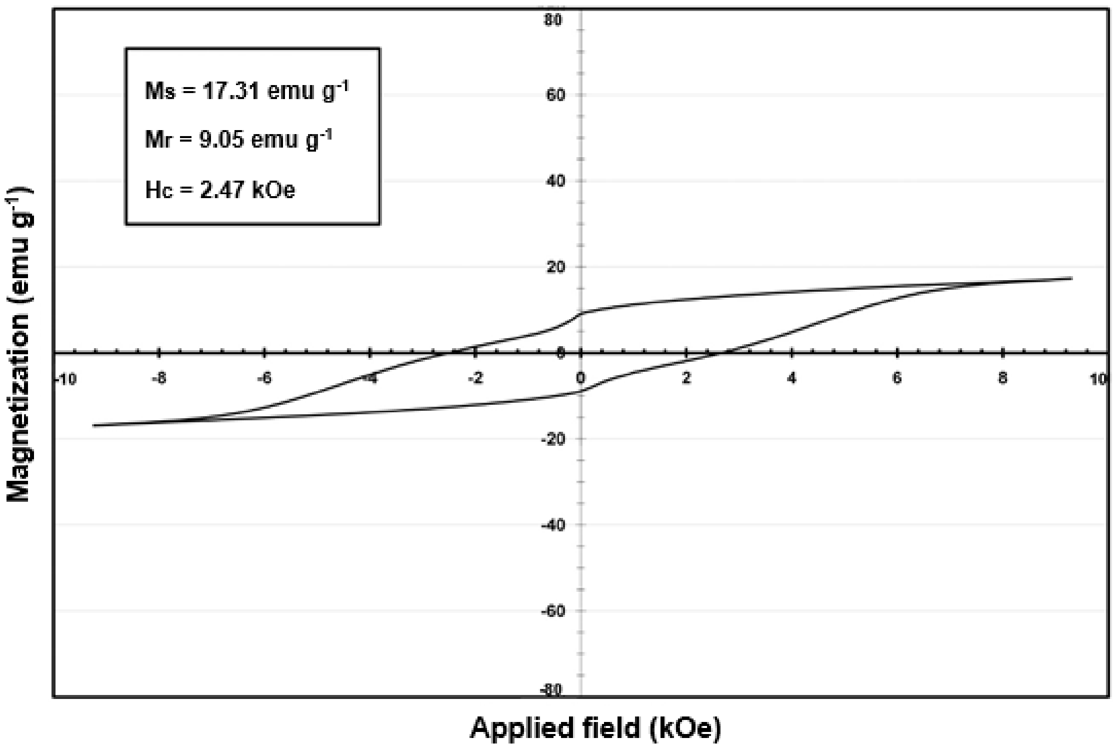

Figure S7. The hysteresis curve of the sample was reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc $min^{-1,}$ and time: 30 min) and then calcined at 700ºC.

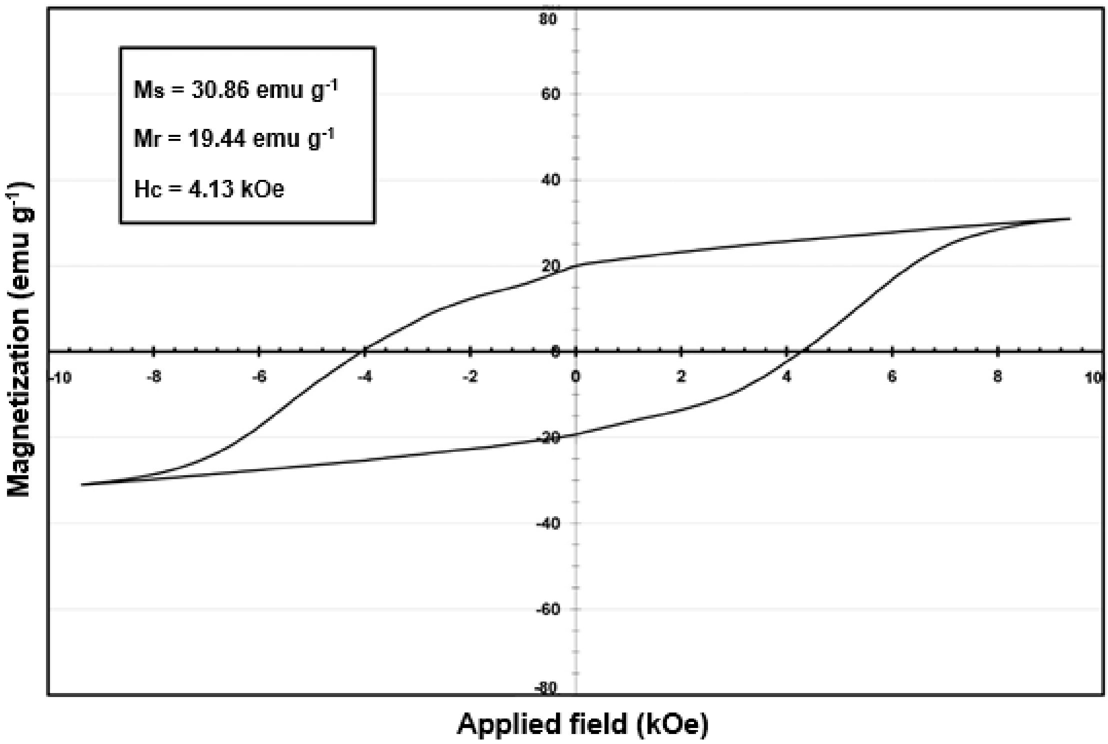

Figure S8. Hysteresis curve of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min) and then calcined at 800ºC.

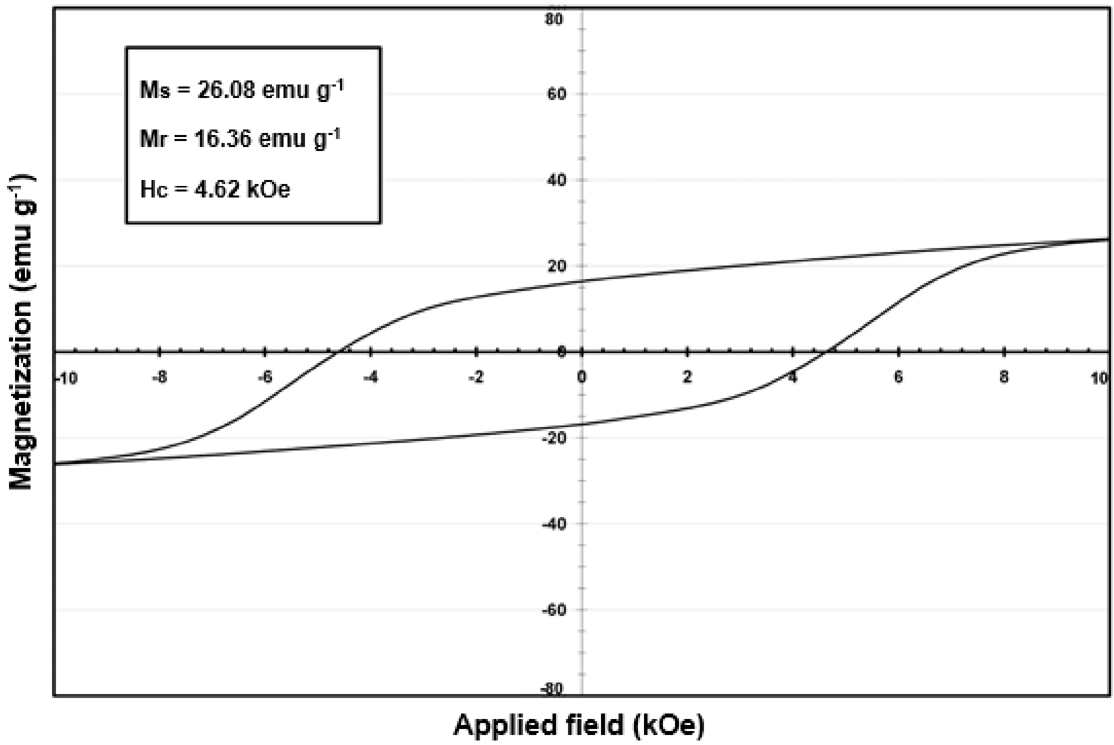

Figure S9. Hysteresis curve of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1}, and time: 30 min) and then calcined at 900ºC.

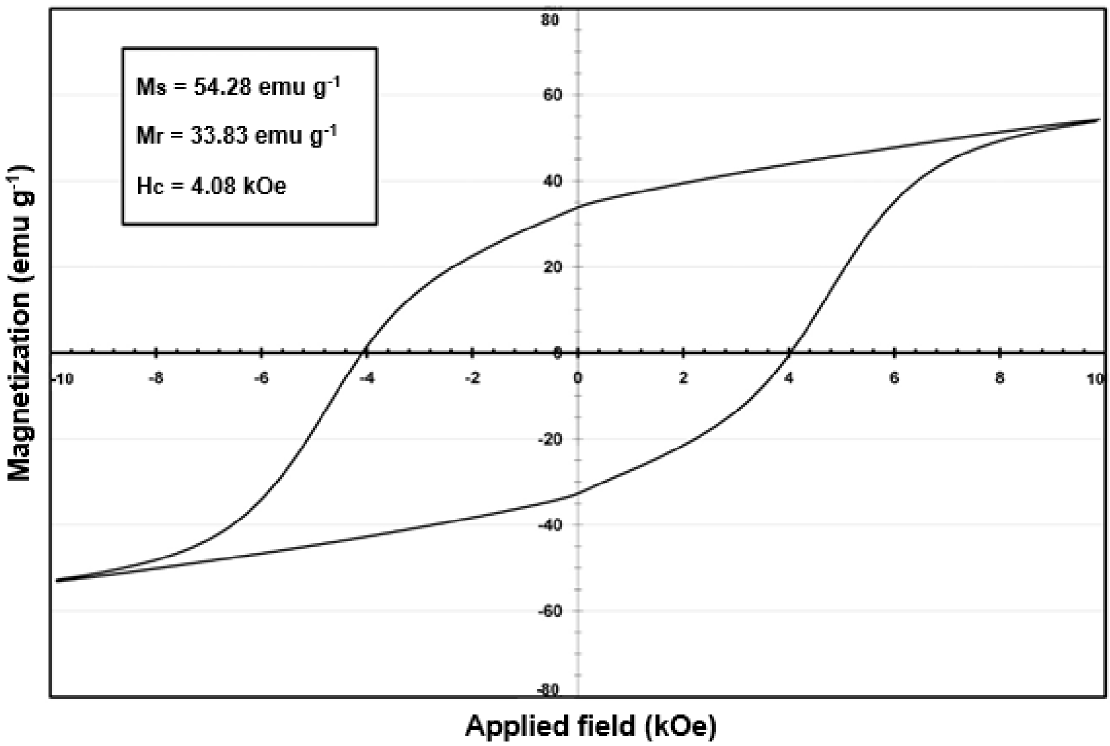

Figure S10. Hysteresis curve of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min) and then calcined at 1000ºC.

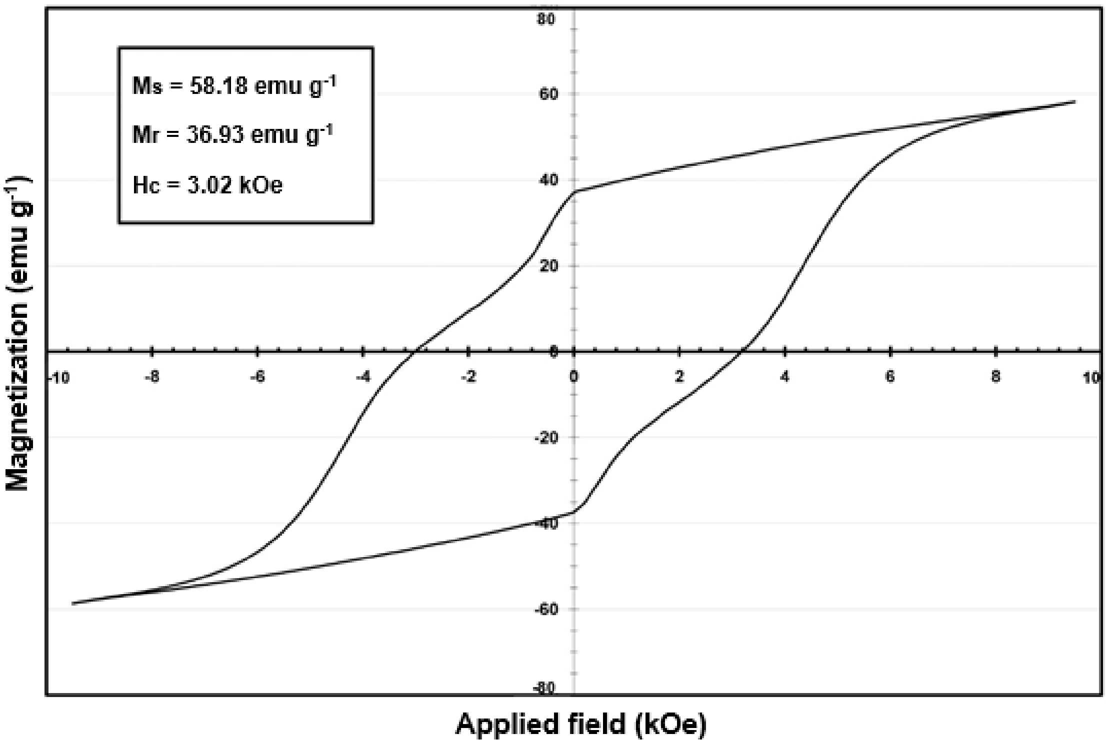

Figure S11. Hysteresis curve of sample reduced under the best conditions (temperature: 950°C, gas flow rate:15 cc min$^{-1,}$ and time: 30 min) and then calcined at 1100°C.

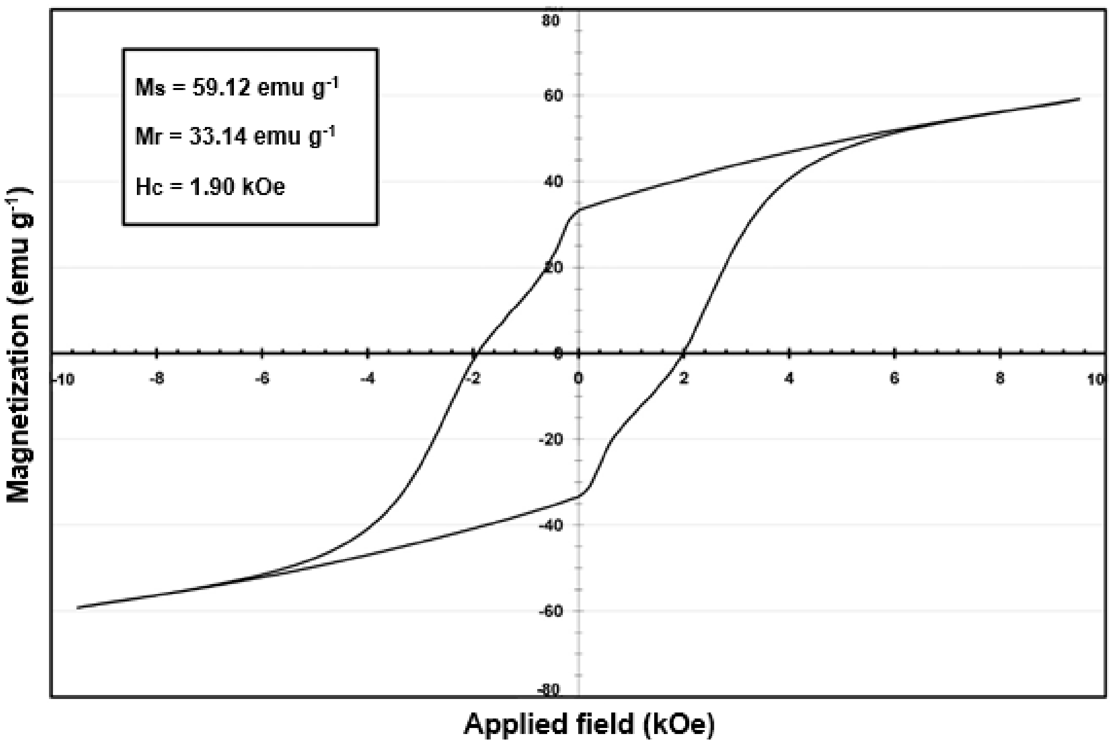

Figure S12. The hysteresis curve of the sample was reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min$^{-1,}$ and time: 30 min) and then calcined at 1200ºC.

5. FE-SEM images of samples

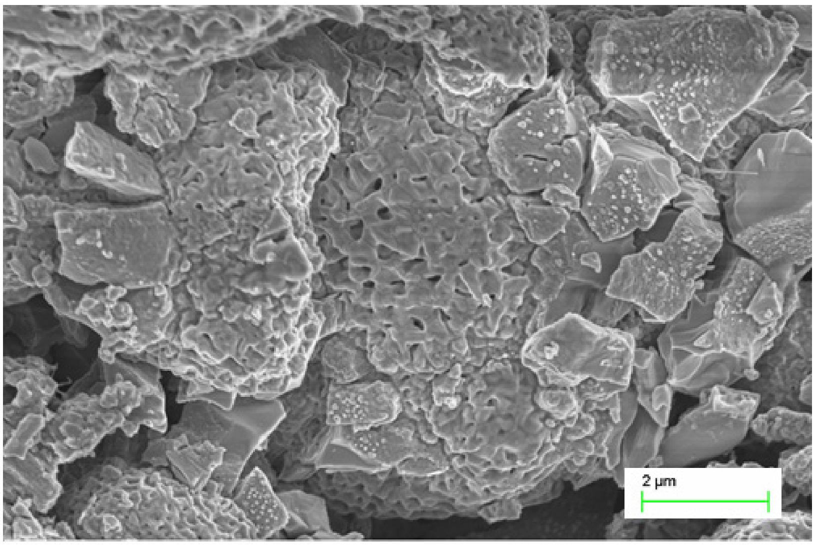

Figure S13. FE-SEM image of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min) and then calcined at 700ºC.

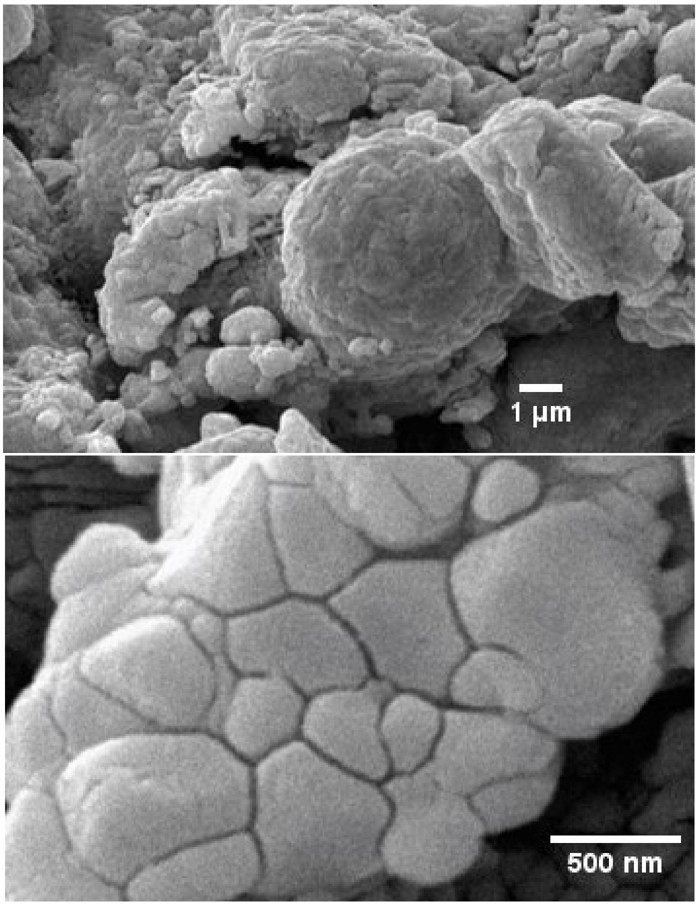

Figure S14. FE-SEM images of the sample reduced under the best conditions (temperature: 950ºC, gas flow rate:15 cc min^{-1} and time: 30 min) and then calcined at 1000ºC for one hour.

6. TEM image of optimum sample

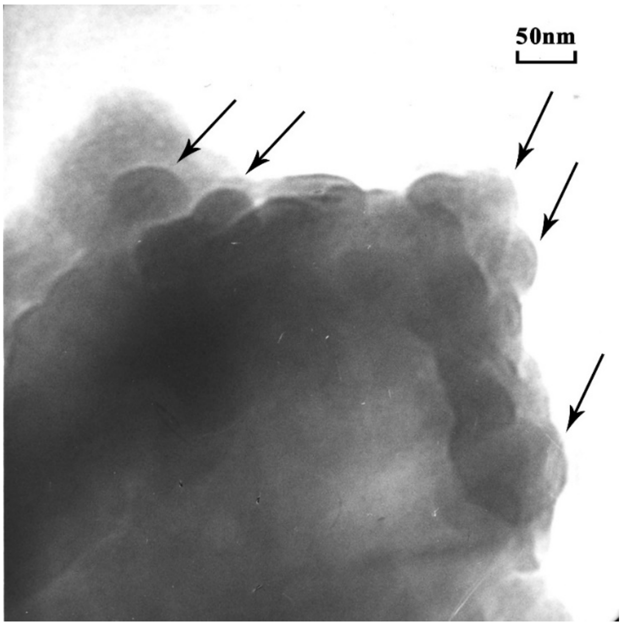

Figure S15. TEM images of the sample reduced under the best conditions (temperature: 950°C, gas flow rate:15 cc min^{-1} and time: 30 min) and then re-calcined at 1000°C for one hour [2].

References

[1] Kürşat, İ. Ç. İ. N., ÖZTÜRK, S., ÇAKIL, D. D., & SÜNBÜL, S. E. (2021). Mechanochemical synthesis of SrFe12O19 from recycled mill scale: Effect of synthesis time on phase formation and magnetic properties. *Journal of Alloys and Compounds*, *873*, 159787.

[2] Dehghan, R., & Ebrahimi, S. S. (2014). Optimized nanocrystalline strontium hexaferrite prepared by applying a methane GTR process on a conventionally synthesized powder. *Journal of magnetism and magnetic materials*, *368*, 234-239.