TECHNICAL UNIVERSITY OF CLUJ-NAPOCA

ACTA TECHNICA NAPOCENSIS

Series: Applied Mathematics, Mechanics, and Engineering
Vol. 65, Issue Special IV, December, 2022

CHI-IRON CARBIDE, AN EMERGING MATERIAL

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Abstract: *The tremendous development of technologies as well as the relentless demand for the performance of materials and devices have resulted in new ways of using certain compounds. In this paper, χ-Fe_5C_2, originally known in the fields of metallurgy and catalysis, is offered as an example. It is shown, thanks to recent data from the literature, this carbide is the subject of intense research extending to other fields to meet new challenges in particular as nanoparticles. Illustrations of the past decade inform about the role of this carbide in clean energy, health and environment. They testify to a capacity for innovation that makes this carbide an emerging material.*
Key words: *: χ-Fe_5C_2, Materials synthesis, Properties, Nanoparticles, Nanostructure, Applications.*

1. INTRODUCTION

This paper deals with chi-iron carbide, also called Hägg carbide, χ-Fe_5C_2 or simply Fe_5C_2. It is a broadly studied member of iron carbides (ICs). Originally chi-iron carbide was detected for almost a century now, as a transient iron carbide during the tempering of steels or as a by-product of the transformation of iron used as a catalyst in Fisher-Tropsch synthesis (FTS) for hydrocarbon production. The first powder diffraction data was reported by Hägg in 1934 [1]. Until now, chi-iron carbide has never been found on earth but it was recently identified in meteorites under the mineral name edscottite [2].

Here, the paper is presented in six sections. The first one concerns the main features of this carbide in terms of atomic structure, principal properties and synthesis. The second one deals with its role in the field of clean energy. The third one focusses on its role in the field of medicine and the field of environment. The last ones draw the conclusion and gives the references.

2. MAIN FEATURES OF FE_5C_2

They concern its atomic structure with main resulting properties and its synthesis.

2.1 Atomic structure and main properties

Its unit cell, first established in 1962 [3], is monoclinic with the C2/c space group. It contains twenty atoms of iron and eight atoms of carbon (see Fig. 1.) leading to the Fe_5C_2 formula. Although sometimes subject to discussion [4], it is the atomic structure used by most authors. The lattice parameters are around a=1.1563 nm, b=0.4573 nm, c=0.5058 nm and β=97.75°.

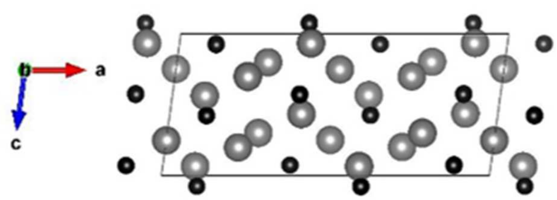

Fig. 1. Fe_5C_2 unit cell modelled through the VESTA software [5]. Large grey balls for Fe atoms and small black for C carbon atoms. Crystallographic data of [3].

From the nature of the atoms, their binding and their arrangement, derive two main properties that are the magnetic and the catalytic properties. There will be exploited, individually or together, for their excellence in chi-iron carbide most often used in the form of nanoparticles (NPs). Indeed, it is true that chi-iron carbide belongs to the class of ICs recognized in 2019 as 'Magic materials with magnetic and catalytic properties' [6].

2.2 Synthesis

There is always a strong demand to use Fe_5C_2 as a pure phase. Different means can synthesize it as NPs. Table 1 gives a chronological list of the processes developed to obtain a pure chi-iron carbide phase for different scientific fields.

About the processes:

- Solid-gas reactions include reactions of $Fe(CO)_5$ iron carbonyl with a CO/H_2 mixture [7], of α-Fe_2O_3/Fe_3O_4 iron oxides mixture with CO [8] and of β-Fe_2O_3 with CO [9].
- Solid-solid reactions concern reaction of α-Fe with g-Fe_3N_4 [10]
- Solid-liquid reactions include reaction of amorphous Fe NPs in oleylamine (OAm) [11] and mechanosynthesis of α-iron in a medium of liquid hydrocarbons [12].
- Wet chemical synthesis [13-15] deal with reactions of $Fe(CO)_5$ iron carbonyl with octadecylamine in the presence of surfactants.

Table 1

Synthesis methods for pure chi-iron carbide phase.

Process	Scientific fields	Year	Ref
Sol.-gas reaction	Magnetism	1971	[7]
Sol.-gas reaction	Magnetism	1990	[8]
Wet chem. synthesis	Catalysis FTS	2012	[13]
Sol.-sol. reaction	FTS Catalysis	2015	[10]
Sol.-liq. reaction	Magnetism	2015	[12]
Sol.-gas reaction	Electrochem	2017	[9]
Wet chem. synthesis	FTS	2017	[14]
Sol.-liq. reaction	Magnetism	2019	[11]
Wet chem. synthesis	FTS Catalysis	2020	[15]

About the involved scientific fields, magnetism and catalysis dominate in links with previously cited properties. The timeline (column 3) shows, on the one hand, that emphasis has been placed on catalytic applications for the past ten years (especially in FTS, where chi-iron carbide is considered as the most active catalyst).

On the other hand, the interest in magnetic applications seems disappears around 1990 (presumably due to the end of magnetic tapes) to reappear around 2015 (probably due to new medical needs).

Efforts made in synthesis of pure Fe_5C_2 have made it possible to develop new Fe_5C_2-based materials as magnetic/catalytic nanostructures suitable for diverse applications in emerging fields [16].

3. FE$_5$C$_2$ IN CLEAN ENERGY

This section mainly gives information on the different functionalized Fe_5C_2 NPS that can bring progress in the field of clean energy as catalysts and electrodes.

3.1 New catalysts for FTS

FTS has received renewed interest in recent years due to the necessity to decrease global dependency on fossil fuels. As Fe_5C_2 plays an important role in this process [17 and references therein], great effort has been made to develop more efficient Fe_5C_2-based catalysts by tuning its catalytic properties through its design [18].

As illustration, table 2 gives a brief timeline overview of Fe_5C_2-based catalysts developed these last years in the aim to increase their selectivity toward different products (olefins, gazoline, alcools) or different process temperatures (low temperature or high temperature).

The meaning of catalyst names is as follows: Fe_5C_2@C for carbon encapsulated Fe_5C_2 NPs [19], Fe_3O_4@Fe_5C_2 for Fe_5C_2 encapsulated Fe_3O_4 core-shell NPs [20], Fe_5C_2/Co for heterostructured NPs made of Fe_5C_2 and Co [21], *Fe_5C_2@CMK-3* for ordered mesoporous carbon containing numerous and well-dispersed Fe_5C_2 nanoparticles [22], Cu-0-chi-Fe_5C_2 for Cu^0–χ-Fe_5C_2 binary catalysts [23], O-décor. Fe_5C_2 for Fe_5C_2 decorated with oxygen (photothermal catalyst) [24], Fe_5C_2/NCT for Fe_5C_2 loading on tunable N-doped carbon (photothermal catalyst) [25], Mn1K-FeC for Mn- and K-modified Fe_5C_2 NPs [26].

Table 2

Fe_5C_2-based FTS catalysts for special tunings.

Catalyst name	Special tunings	Year	Ref
Pure Fe_5C_2	Olefins	2015	[10]
Fe_5C_2@C	High-temp.	2015	[19]
Fe_3O_4@Fe_5C_2	Olefins	2016	[20]
Fe_5C_2/Co NPs	Low-temp.	2017	[21]
Fe_5C_2@CMK-3	Gasoline	2017	[22]
Cu-O-chi-Fe_5C_2	Higher alcools	2017	[23]
O-decor. Fe_5C_2	Lower olefins	2018	[24]
Fe_5C_2/NCT	Ligh olefins	2022	[25]
Mn1K-FeC	Higher alcools	2022	[26]

3.2 New electrodes and nanomagnets for energy conversion or storage

In energy conversion, fuel cells, metal-air batteries, water splitting using hydrogen evolution reaction (HER) for hydrogen production have been regarded as possible solutions for safe and convenient power generation applications, requiring to develop new materials such as electro-catalysts.

In this context, table 3 presents new Fe_5C_2-based materials developed these four last years. The first example concerns a soft magnetic N-doped carbon encapsulated (Fe_5C_2-Fe_3C@CN) showing an efficient HER catalytic activity to replace Pt electrode [27].

Table 3

Fe_5C_2-based materials for energy conversion/storage.

Name	Device	Year	Ref.
Fe_5C_2-Fe_3C@CN	HER/water splitting	2019	[27]
Fe/Fe_5C_2@N-C	ORR/ Zinc-air battery	2019	[28]
Fe_2O_3/Fe_5C_2/Fe-N-C	ORR/ Zinc-air battery	2022	[29]
3DM Fe/Fe_5C_2@NC	LIB/PIB	2020	[30]
$BaFe_{12}O_{19}$/Fe_5C_2	nanomagnet	2019	[31]

Two other examples are given for their high performances and superior stability than that of Pt/C in zinc-air batteries. The first one is an oxygen reduction reaction (ORR) electro-catalyst made of Fe/Fe_5C_2 species enveloped in N-doped carbon called Fe/Fe_5C_2@N-C [28]. The second one concerns the fabrication of new Fe_2O_3/Fe_5C_2/Fe-N-C composites composed of Fe_2O_3/Fe_5C_2 NPs and Fe,N-doped carbon species [29].

In energy storage, the demands for portable electronic devices and electric vehicles are constantly increasing. To address this, great efforts are focused on the development of high energy density and long-life renewable energy batteries.

Efficient lithium/potassium-ion batteries (LIBs/PIBs) require high-capacity anode materials whose synthesis remains a big challenge.

On the one hand, It has been shown that Fe/Fe_5C_2 NPs coupled with nitrogen-doped porous carbon, called 3DM Fe/Fe_5C_2@NC own outstanding lithium/potassium storage properties. It can thus serve as an anode material [30].

On the other hand, to replace the NdFeB- and SmCo-based magnets, it is extremely essential to develop exchange-coupled nanomagnets with rare-earth free hard/soft magnetic phases to apply energy-related devices. It is a heavy task but a conclusive test was obtained with $BaFe_{12}O_{19}$/Fe_5C_2 composites fabricated through cryogenic ball milling [31].

4. FE_5C_2 IN MEDICINE AND IN ENVIRONMENT

4.1 In medicine

Due mainly to their high magnetism, Fe_5C_2 nanoparticles play an important role among the iron carbide NPs used for biomedical applications [32], tending to replace iron oxyde NPs dominantly used. In a very recent review [33], iron carbide nanostructures of which Fe_5C_2 is a part are described as an 'emerging material for tumor theranostics', involved in both diagnosis and therapy.

Among the diagnostic means, magnetic resonance imaging (MRI) is one of the most widely used methods for detecting tumors. To detect them as early as possible, MRI requires increasingly effective contrast agents, including Fe_5C_2 NPs for their high contrast and their ability to target tumors. Table 4 summarizes Fe_5C_2-based nanostructures as contrast agent to diagnose different tumor thanks to surface modifications.

Table 4

Fe_5C_2-based materials for tumor diagnosis in MRI.

Contrast agent	Tumor	Year	Ref.
Fe_5C_2 NPs	U87MG	2014	[34]
Fe_5C_2-Z$_{HER2:342}$ NPs	SK-OV-3	2014	[35]
Casein-coated Fe_5C_2	Of liver	2015	[36]
Fe_5C_2-GOD@MnO_2	U14	2018	[37]
Fe_5C_2@Fe_3O_4 NPs	4T1	2019	[38]

Concerning the different names of the contrast agents, Fe_5C_2-ZHER2:342 NPs means that Fe_5C_2 NPs are new proteins coated; Fe_5C_2-GOD@MnO_2 indicates a core-shell nanostructure with a glucose oxidase (GOD)-loaded Fe_5C_2 core and a manganese dioxide (MnO_2) shell; Fe_5C_2@Fe_3O_4 is for Fe_5C_2 core with a magnetite shell.

Among tumor therapy means, table 5 summarizes Fe_5C_2-based nanodrugs able to kill specific tumor cells with low cytotoxicity in photothermal therapy (PTT), chemotherapy (chemo), chemodynamic therapy (CDT) and photodynamic therapy (PDT). Some Fe_5C_2-based materials developed for tumor diagnosis in MRI (see Table 4) serve as nanodrugs.

It is then possible to ablate tumors under guidance by MRI and PTT without noticeable side effects. Others Fe_5C_2-based materials are developed with new surface modifications for the needs of therapy. These are Fe_5C_2-BSA-DOX NPs and Fe_5C_2-BSA-AIPH/PCM NPs with BSA for bovin serum albumin, DOX for doxorubicin drug, ALPH for a polymerization initiator loaded on the surface of Fe_5C_2-BSA NPs and PCM for a higher drug load and controlled heat-stimulated drug release.

Table 5

Fe_5C_2-based materials for tumor therapy.

Nanodrug	Tumor	Therapy	Year/Ref
Fe_5C_2-ZH$_{ER2:342}$ NPs	SK-OV-3	PTT	2014 [35]
Fe_5C_2-BSA-DOX NPs	SK-OV-3	PTT Chemo	2016 [39]
Fe_5C_2-GOD@MnO_2	U14	CDT	2018 [37]
Fe_5C_2-BSA-AIPH/PCM NPs	U14	PTT & PDT	2018 [40]
Fe_5C_2@Fe_3O_4	4TI	CDT	2019 [38]

4.2 In environment

Chi-iron carbide acts in environment remediation. Table 6 illustrates this. It was shown in 2015 that Fe_5C_2 NPs with a thin carbon shell coating have a photothermal effect and possess reusable antibacterial properties [41] useful for disinfection of *E. coli* and *S. aureus* in water.

In the case of drug detection, it was proven in 2017 that high-purity chi-carbide nanoparticles can serve as promising electrocatalyst for voltametric detection of the antibiotic metronidazole [9]. In the case of waste treatment to remove pollutants as heavy metals in water, novel and high efficient magnetic alginate beads containing Fe_5C_2@SiO_2 core-shell (NPs) were synthesized and applied to remove Cu (II) ions from water [42] in 2018. Finally, in the case of mitigation of electro-magnetic pollution (EM), a magnetic-dielectric Fe/Fe_5C_2@NC composite as an efficient microwave absorbent [43] was very recently developed in the field of electromagnetic wave adsorption.

Table 6

Fe_5C_2-based materials for environment remediation.

Name	Action	Year	Ref.
Fe_5C_2 @C NPs	Antibacterial disinfection	2015	[41]
Fe_5C_2 NPs	Antibiotic detection	2017	[9]
Fe_5C_2@SiO_2 NPs	Heavy metals removal	2018	[42]
Fe/Fe_5C_2@NC composite	Microwave absorbent	2022	[43]

5. CONCLUSION

This article traces the path of a compound discovered fortuitously almost a century ago.

It is shown that many Fe_5C_2-based materials have been developed as NPs over the past ten years in connection with societal issues such as clean energy, health and environment. These materials have made it possible to improve applications and devices. Highlights are following:

In the field of clean energy, new Fe_5C_2-based catalysts in the aim to increase their selectivity toward different products, new Pt-free electrodes and new rare-earth free nano-magnets with a performance gain and a lower cost;

In the field of health and medicine, its recent role as contrast agent for tumor diagnosis and as nanodrug for tumor therapy to replace iron oxides NPS more efficiently;

In the field of environment, its recent role in antibacterial disinfection, antibiotic detection, heavy metals removal and in mitigation of electromagnetic pollution.

The use of Fe_5C_2 as NPs globally leads to progress in many applications and devices. This makes it possible to demonstrate that this carbide is becoming an emergent material.

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CARBURA DE FIER-HI, UN MATERIAL EMERGENT

Dezvoltarea deosebită a tehnologiilor, precum și cererea continuă de creștere a performanțelor materialelor şi dispozitivelor, au determinat apariția unor noi modalități de utilizare a anumitor compuși. În această lucrare, χ-Fe5C2, cunoscut inițial în domeniile metalurgiei și catalizei, este prezentat ca un exemplu de asemenea compus. Pe baza rezultatelor recente prezentate în literatură, se arată că această carbură constituie obiectul unor cercetări care se extind și în alte domenii, pentru a face față noilor provocări, în special a celor referitoare la nanoparticule. Exemplele din ultimul deceniu evidențiază rolul pe care îl poate avea carbura hi pentru asigurarea unei energii curate, pentru sănătate și pentru mediul înconjurător. Ele dovedesc totodată o capacitate de inovare care face din această carbură un material emergent.

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