SiC Synthesis Using Natural Mg-Silicates

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Abstract:

The silicon carbide powders are prepared by carbothermal reduction of domestic mineral resources (white and brown sepiolite), at relatively low temperatures (1200 – 1600°C). Carbothermal reduction process is greatly influenced by chemical composition of sepiolites and type of carbon used. Obtained SiC powders consist of fine β-SiC particles and did not retain the fibrous morphology of sepiolites. Catalytical influence of Fe is attributed to formation of iron-silicide and its potentionaly important role in removal of Mg-species, i.e. reduction of Mg_2SiO_4 and MgO into $Mg_{(g)}$.

Keywords: *SiC; Carbothermal reduction; Sepiolite; Minerals; Silicates.*

1. Introduction

Silicon carbide is an important structural material because of its unique combination of properties, such as high temperature strength, thermal shock resistance and resistance to wear and corrosion [1]. Thus, SiC has been a major candidate material for widespread use in engineering applications. The synthesis of pure SiC powder is one of the important factors in obtaining dense SiC ceramics [2]. Although the quality and performance are important, the cost of mass production should be the key factor in the commercialization of SiC ceramics. The most promising technique for obtaining variety of non-oxides products with important technical uses is carbothermal reduction (CRR) reaction [3, 4].

This reaction involves reduction of oxygenated materials (for example silica - SiO_2) with reducing agent (carbon) under an inert atmosphere, at the temperatures usually higher than 1600°C. This liberates Si or SiO in gaseous form, which further reacts with excess carbon to form SiC following the general reaction [5, 6]:

$$SiO_2 + 3C \Rightarrow SiC + 2CO_{(g)} \tag{1}$$

Formation of the final product is more complex than described by the above equation because of many intermediate stages [5, 6].

CRR offers the possibility of an economically attractive production route for SiC,

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basing on naturally occurring materials. Many authors have studied the formation of SiC powders from the raw materials, such as high purity quartz sand [5, 7, 8]. Among the raw materials that may be used for SiC powder production, sepiolite has some advantages such as high specific surface area, high silica content and low price. Several papers were published with the similar subject, i.e. production or sintering of Si_3N_4 powders from carbothermally reduced sepiolite [9, 10].

Sepiolite is a naturally occurring mineral (magnesium-silicate) with fibrous morphology, whose structure is composed of two bands of silica tetrahedrons linked by magnesium ions in octahedral coordination, thus forming open channels of fixed dimensions running parallel to the chains [11]. Such a high surface area is beneficial for the synthesis of SiC powder by carbothermal-reduction reaction, especially when reducing agent is introduced via liquid solution. However, by our best knowledge, no work has yet been done on synthesis of SiC by using sepiolite or other Mg-silicates as Si source.

In this paper, CTR method has been used to produce SiC powder from the two sepiolites (as SiO_2 source) and three carbon precursors (saccharose, activated carbon, novolac resin) as reducing agents.

2. Experimental procedure

A two sepiolites of Serbian origin (chemical formula: $Mg_4Si_6O_{15}(OH)_2\cdot6H_2O$) and carbon obtained from different precursors (saccharose, activated carbon, novolac resin), were used for production of SiC powders. Chemical composition of as-received sepiolites is shown in Tab. I, together with the expected composition calculated from the chemical formula of sepiolite. Weight loss at $800^{o}C$ corresponds to the loss of water i.e. dehidratation of sepiolite [11]. Carbon precursors used are: Saccharose, $C_{11}H_{22}O_{11}$, Alfa Aesar, (p.a., heavy metals<5ppm, Fe<5ppm); Novolac resin ("Bakelite", 6109FP); Activated carbon ("Jacobi", Aquasorb HS, inorganic residue max. 0.8 wt.%; AC - in further text). Sepiolite and activated carbon powders were dried ($110^{o}C$, 2h) before preparation of samples in order to remove any moisture.

Tab. I Chemical composition of sepiolites

Wt. %	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	NiO	CuO	Cr_2O_3	TiO_2	Other elements (Mn, Ba, Co, Sr, Zn, Zr, Ni, etc.)	$800^{o}C$ wt. loss
Sepiolite A (brown)	53.72	0.72	2.01	23.14	0.12	0.10	0.09	0.06	0.06	0.05	0.08	<0.005	19.80
Sepiolite B (white)	50.22	-	0.01	27.17	0.07	0.08	0.02	-	0.09	-	-	<0.005	22.20
Expected composition	55.70			24.90									19.40

C/SiO_2 molar ratio in all samples was 7:1, calculated in respect of carbon residue at $800^{o}C$, in order to ensure the presence of an excess of carbon [12]. Sepiolite/carbon samples

made from saccharose and novolac resin were prepared by following procedure: appropriate amounts ($\approx$2g) of saccharose/novolac are dissolved in water/ethanol (50ml) and then mixed with previously dried sepiolite powder ($\approx$1g, 383K, 2h). Excess liquid is removed by gentle heating at 60°C. Polymerization of the samples was conducted by slowly raising the temperature to 180°C in several steps. Samples were then carbonized up to 800°C (2 $^\circ$C /min, Ar flow atmosphere) and then heat-treated at 1200 $^\circ$C - 1600 $^\circ$C in "Astro" graphite furnace (graphite crucibles, 1h soaking time, Ar flow atmosphere, heating rate = 40 $^\circ$C /min). The argon flow was kept during cooling until room temperature.

Structural analysis of powdered samples was carried out by a Siemens D-500 powder diffractometer. CuKα radiation was used in conjuction with a CuK$_\beta$ nickel filter.
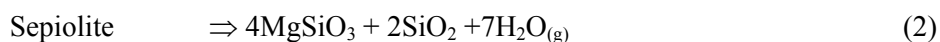
The microstructural study and energy dispersive analysis of X-rays (EDS) were performed on samples with Au coating with VEGA TS 5130mm and JEOL JSM 6460 scanning electron microscopes (SEM). Prior to SEM/EDS analysis, samples are oxidized in air at 600 $^\circ$C (2h), in order to remove the residual (unreacted) carbon that, if not removed, makes SEM/EDS analysis rather difficult.

Chemical composition of dried sepiolites was obtained by XRF analysis with Axios – PANalytical instrument.


3. Results and discussion

Phase content of CTR reaction products, as observed from XRD patterns of sepiolite/carbon samples heat-treated at 1200 $^\circ$C -1600 $^\circ$C, can be seen in Tab. II. Temperatures where pure SiC phase appears for the first time are marked in bold. We use the term "pure SiC phase" for samples containing Fe_3Si and amorphous carbon also, because Fe_3Si phase can be easily removed by leaching in acids, whilst free carbon is easily removed by oxidation in air [13]. SiC is identified in all samples as β-form (JCPDS number 29-1129). Forsterite (Mg_2SiO_4, 34-0189), Enstatite ($MgSiO_3$, JCPDS number 11-0273), Periclase (MgO, JCPDS number 43-1022) and Suessite (Fe_3Si, JCPDS number 35-0519) are also identified in some samples.

When pure sepiolite is heated up to 1400 $^\circ$C , enstatite ($MgSiO_3$) is formed together with cristobalite (SiO_2) as was observed before [9]. Obviously, forsterite (Mg_2SiO_4) formation, as observed from XRD patterns shown in Figs. 1–6, is linked with presence of carbon and these processes can be described by considering the following steps:

$$\text{Sepiolite} \Rightarrow 4MgSiO_3 + 2SiO_2 + 7H_2O_{(g)} \tag{2}$$

$$2MgSiO_3 + 3C \Rightarrow Mg_2SiO_4 + SiC + 2CO_{(g)} \tag{3}$$

Reduction of forsterite into the MgO and latter disappearance of MgO with temperature increase, as observed from XRD patterns of samples shown in Figs. 2-6, can be described by two-step reduction:

$$Mg_2SiO_4 + 3C \Rightarrow SiC + 2MgO + 2CO_{(g)} \tag{4}$$
$$MgO + C \Rightarrow Mg_{(g)} + CO_{(g)} \tag{5}$$
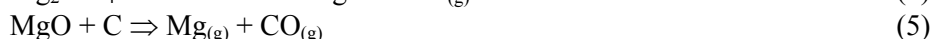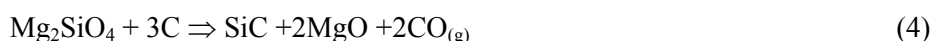
Disappearance of forsterite without MgO formation, as observed in XRD patterns of sample prepared with sepiolite A and novolac resin (Fig. 1), can be explained by forsterite reduction in presence of an excess of carbon, as proposed by Kurt and Davies [10]:

$$Mg_2SiO_4 + 3C \Rightarrow SiO_{(g)} + 2Mg_{(g)} + 3CO_{(g)} \tag{6}$$

According to this, Mg was evaporated together with CO and swept away by the argon flow. Gaseous SiO may react with carbon and produce SiC or also be swept away by argon flow.

However, question is why the reaction (6) is not occuring in all other samples with the same C/Si ratio, i.e. where excess carbon is also presented (Figs. 2-6).
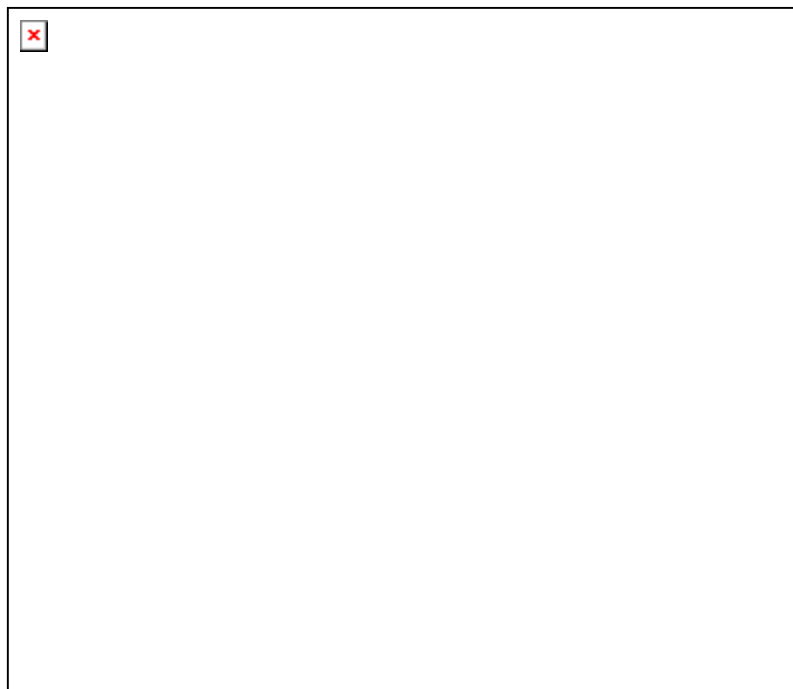

Fig. 1. XRD patterns of Sepiolite A / novolac samples heat treated at: a) 1200 °C; b) 1300 °C; c) 1400 °C; d) 1500 °C; e) 1600 °C.
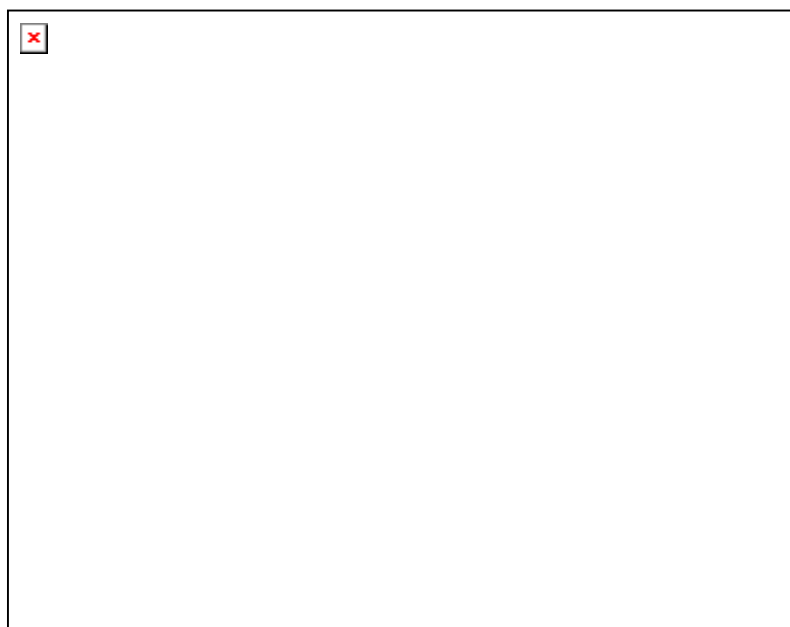

Fig. 2. XRD patterns of Sepiolite A / saccharose samples heat treated at:a) 1200 °C; b) 1300 °C; c) 1400 °C; d) 1500 °C; e) 1600 °C.
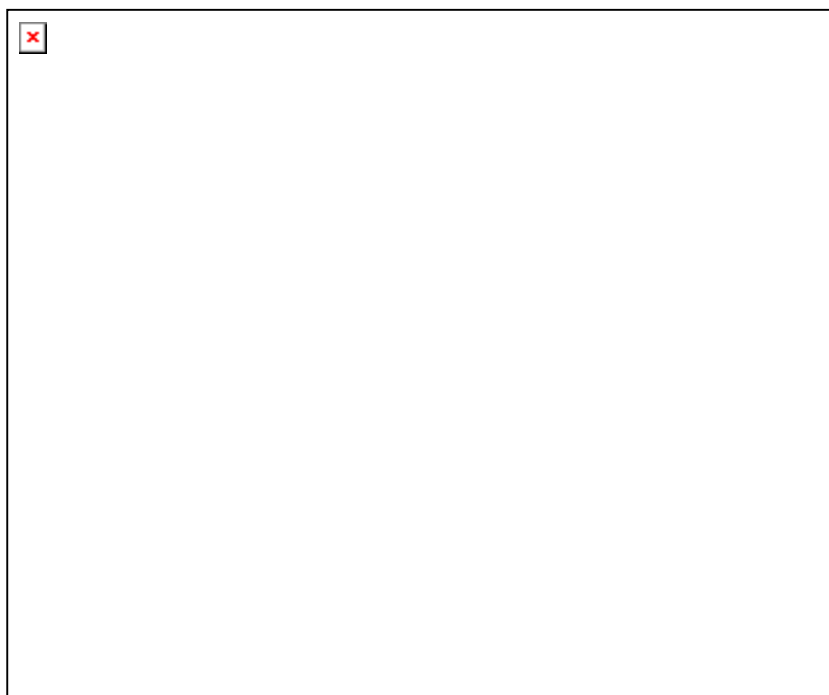
Fig. 3. XRD patterns of Sepiolite A / AC samples heat treated at:a) 1200 ^{o}C; b) 1300 ^{o}C; c) 1400 ^{o}C; d) 1500 ^{o}C; e) 1600 ^{o}C.

Fig. 4. XRD patterns of Sepiolite B / novolac samples heat treated at:a) 1200 ^{o}C; b) 1300 ^{o}C; c) 1400 ^{o}C; d) 1500 ^{o}C; e) 1600 ^{o}C.(marks "3x" and "10x" means that the intensity of the pattern was three i.e. ten times increased for clarity)

Suessite i.e. iron-silicide phase (Fe$_3$Si) is formed only in samples prepared with sepiolite A, which contains significant amount of Fe (Tab. I). Absence of suessite in samples prepared with sepiolite B is due to the absence of Fe in the starting material (Tab. I).

Although it is well known that iron-silicides are commercially used for $MgO \rightarrow Mg_{(g)}$ reduction [14], amount of iron-silicides formed in our samples is obviously not big enough to significantly influence the course of CRR reaction, because in both Fe-containing sepiolite A/carbon samples and in Fe-free sepiolite B/carbon samples, pure SiC samples/powders are generally obtained only at 1600 ^{o}C (with the exception for sepiolite A/novolac sample). Type of carbon precursor used also seems to have no influence, as one may conclude from data presented in Tab. II.

Tab. II. Phase composition of samples heat-treated at different temperatures

| Sample	1200 ^{o}C	1300 ^{o}C	1400 ^{o}C	1500 ^{o}C	1600 ^{o}C
Sepiolite A + saccharose			SiC, F, S	SiC, MgO, S	SiC, S
Sepiolite A + novolac		SiC, E, F, S	SiC, S	SiC, S	SiC, S
Sepiolite A + AC	E, SiC		SiC, F, S	SiC, MgO, S	SiC, S
Sepiolite B + saccharose		E, SiC	SiC, F, E	SiC,	SiC
Sepiolite B + novolac			SiC, F, E	MgO	SiC
Sepiolite B + AC			SiC, F		SiC

Legend: E-enstatite; F-forsterite; S-suessite;

Fig 5. XRD patterns of Sepiolite B / saccharose samples heat treated at:
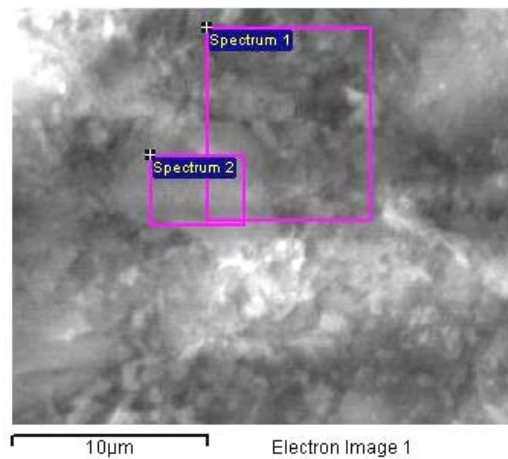   a) 1400 ^{o}C; b) 1500 ^{o}C; c) 1600 ^{o}C.

Fig. 6. XRD patterns of Sepiolite B / AC samples heat treated at:a) 1400 ^{o}C; b) 1500 ^{o}C; c) 1600 ^{o}C.
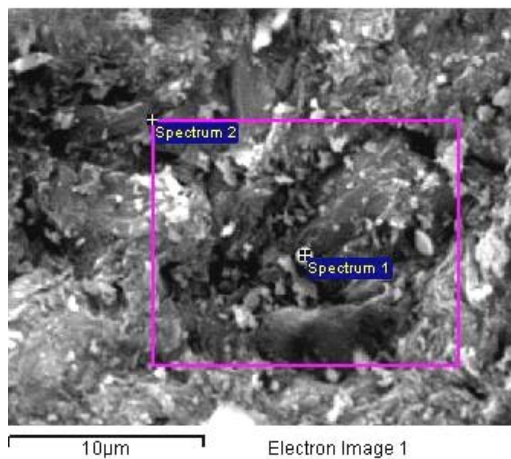
Wt.%	O	Mg	Al	Si	Fe	Total
Spectrum 1	54.04	16.11	0.68	28.20	0.97	100.00
Spectrum 2	54.67	16.52	0.53	27.57	0.71	100.00

(a)

Wt.%	O	Mg	Al	Si	Fe	Total
Spectrum 1	60.76	5.97	0.80	31.83	0.65	100.00
Spectrum 2	57.60	6.89	0.48	34.31	0.73	100.00

(b)

Fig. 7. SEM/EDS analysis of samples heat-treated at 1200 ^{o}C: a) sepiolite A/saccharose; b) sepiolite A/novolac.

Only exception from this general rule, is previously mentioned sepiolite A/novolac sample, which gives pure SiC already at 1400 ^{o}C and which contains both Fe and carbon obtained from novolac resin. Since in all other samples, pure SiC phase is obtained only at 1600 ^{o}C, it is obvious that neither Fe, nor novolac alone, are capable to produce the pure SiC samples at 1400 ^{o}C, but only when they are both present in the system. This is presumably

done by facilitating reactions (4)-(6) since pure SiC phase can be obtained only after all Mg-containing species (enstatite, forsterite, MgO) are removed from the system. This indicate that only the combination of Fe and novolac presence, is capable of producing pure SiC phase at temperature 200 ^{o}C lower than in all other samples.

This assumption was proved by comparative SEM/EDS analysis of sepiolite A/novolac and sepiolite A/saccharose samples heat-treated at 1200 ^{o}C:

By comparing the results of EDS analysis of samples shown in Fig. 7, it is obvious that Mg content is much lower in sample prepared with novolac resin, than in sample prepared with saccharose. This indicate that carbon obtained from novolac resin, in combination with Fe presence, is truly much more efficient in reducing the Mg-containing species into the Mg$_{(g)}$ than saccharose, even at such a low temperature such is 1200 ^{o}C.
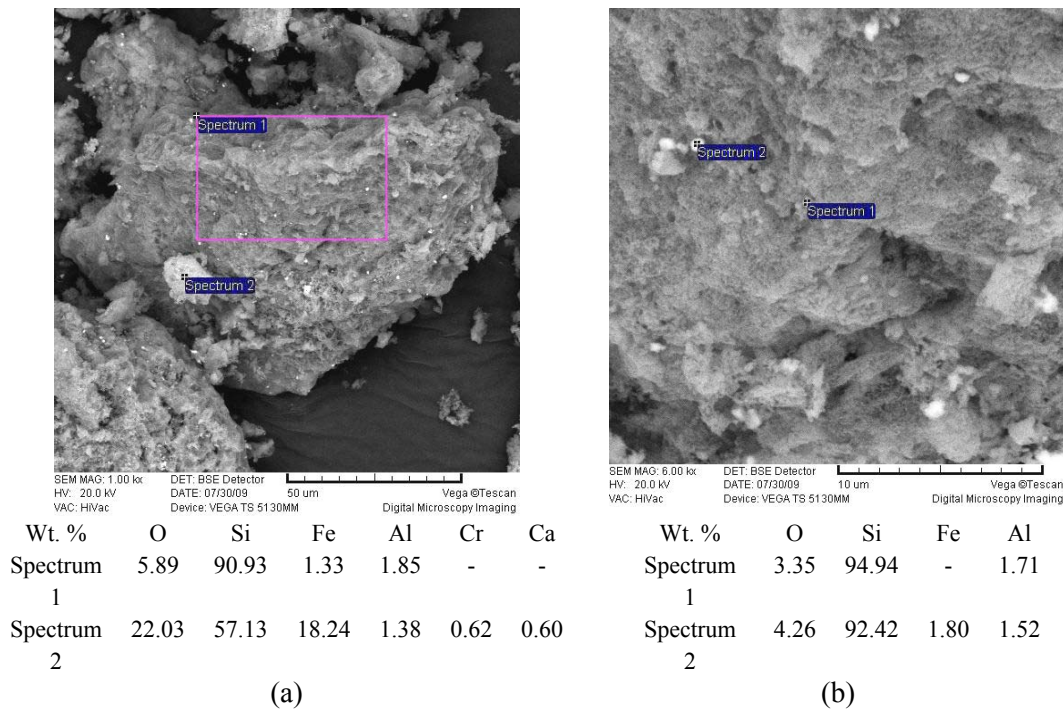

Wt. %	O	Si	Fe	Al	Cr	Ca
Spectrum 1	5.89	90.93	1.33	1.85	-	-
Spectrum 2	22.03	57.13	18.24	1.38	0.62	0.60

(a)

Wt. %	O	Si	Fe	Al
Spectrum 1	3.35	94.94	-	1.71
Spectrum 2	4.26	92.42	1.80	1.52

(b)

Fig. 8. SEM/EDS analysis of sepiolite A/novolac sample heat treated at 1400 ^{o}C

Results of SEM/EDS analysis of sepiolite A/novolac sample heat-treated at 1400 ^{o}C are shown in Fig. 8. As one can see, its chemical composition corresponds to phase composition obtained by XRD analysis (Fig 1c): SiC phase, Fe-rich (Fe$_3$Si) phase and no trace of Mg-containing compounds. By using BSE (Back Scattering Electrons) detector mode, Fe-containing particles became clearly visible, usually as brightly white spheres with ≈1μ in diameter. Since they are smaller than beam focus (≈5μ), their EDS analysis is rather difficult (Fig. 8b, spectrum 2). However, one large light grey particle with high Fe and O content was observed (Fig. 8a, spectrum 2). High O content is probably due to oxygen bonded to Fe$_3$Si phase. This is not surprising, since iron-silicides are commonly used in metallurgy as a "sponge" for oxygen removal from the system [14]. Also, judging by the color, this large particle presumably contains some SiC phase too. Pure SiC phase appears in SEM images as grey porous surface, which consists from agglomerates of very fine particles (≈100nm), with high Si and low O content. Spectrum 1 presented in Fig. 8b shows composition of such SiC domain, free from Fe-containing particles, while spectrum 1 presented in Fig. 8a represents average composition of large area containing both SiC phase an Fe-containing particles. Presence of Ca and Cr in EDS spectra of Fe-rich particle (Fig. 8a, spectrum 2) is also worth

mentioning, since there is no trace of Ca and Cr in SiC-rich domain (Fig. 8a, spectrum 1). We can also conclude that Al is distributed evenly through both Fe-rich and SiC-rich domains. All of these impurities, especially in combination with Si-O and Fe-O species, may produce multiphase eutecticums, which together with iron-silicide (also have a low melting point) can have catalytic influence on the CTR reaction by facilitating the transport of the material. It is quite possible that liquid phase/phases are formed on the grain boundaries, but they are difficult to prove by XRD and SEM/EDS analysis due to the instruments/methods limitation. Only the TEM/EDS analysis could confirm their presence and location in the microstructure, but this is beyond our current capabilities.
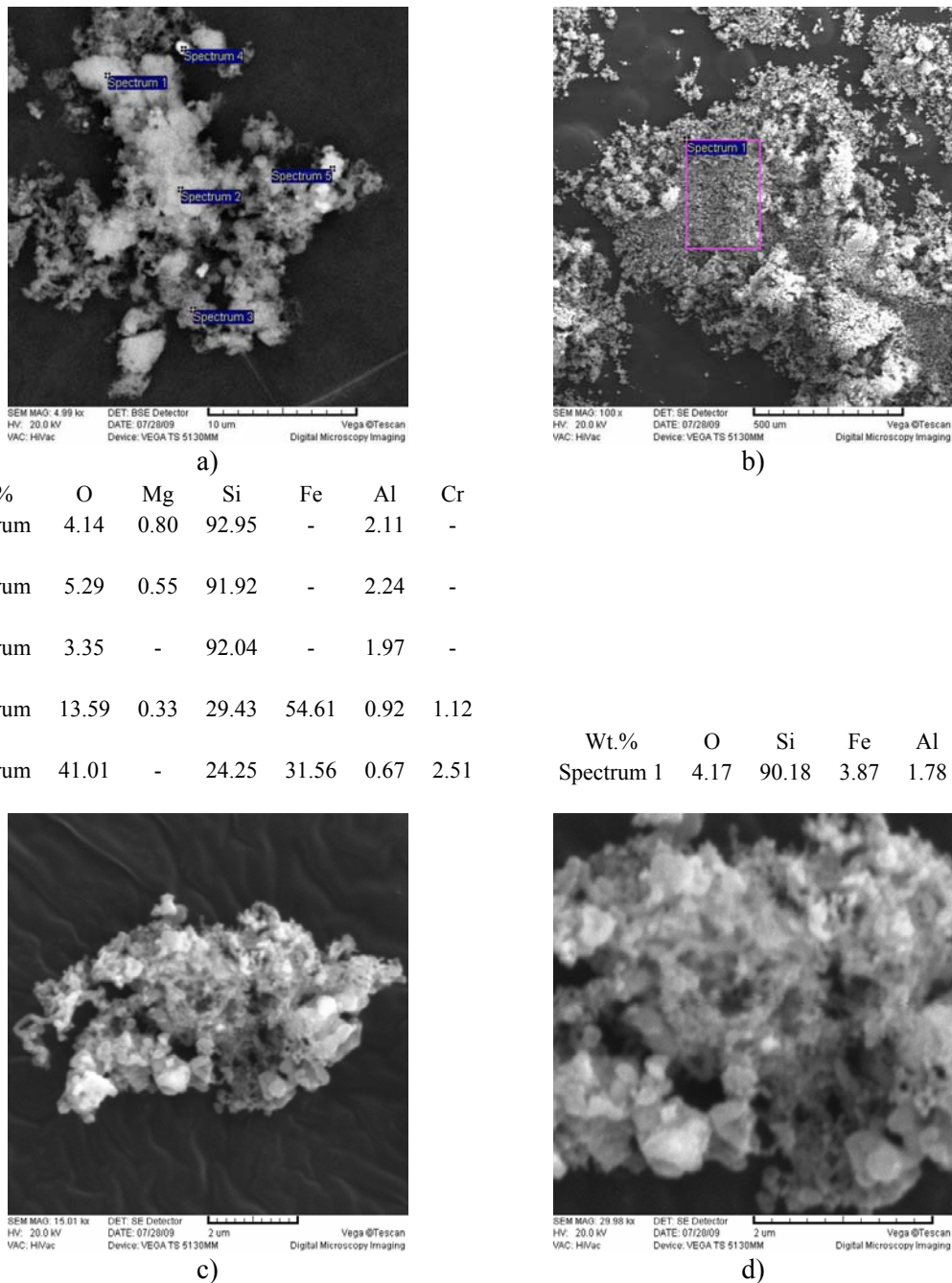

Wt.%	O	Mg	Si	Fe	Al	Cr
Spectrum 1	4.14	0.80	92.95	-	2.11	-
Spectrum 2	5.29	0.55	91.92	-	2.24	-
Spectrum 3	3.35	-	92.04	-	1.97	-
Spectrum 4	13.59	0.33	29.43	54.61	0.92	1.12
Spectrum 5	41.01	-	24.25	31.56	0.67	2.51

Wt.%	O	Si	Fe	Al
Spectrum 1	4.17	90.18	3.87	1.78

Fig. 9. SEM/EDS analysis of sepiolite A/AC sample heat treated at 1600 °C

Results of SEM/EDS analysis of sepiolite A/AC sample heat-treated at 1600°C are shown in Fig. 9. Average chemical composition of large area is given in Fig. 9b. As one can see, its chemical composition corresponds to phase composition obtained by XRD analysis (Fig. 3e): SiC phase, Fe-rich (Fe$_3$Si) phase and only traces of Mg-containing compounds. Detailed EDS analysis is given in Fig. 9a (BSE mode), where Fe-rich and SiC-rich areas can be easily observed, similar to those described for sample sepiolite A/novolac. However, although SEM/EDS analysis shows results/conclusions almost identical to those for sample sepiolite A/novolac at 1400 ^{o}C, it is obvious (Tab. II) that presence of impurities (Al, Cr) and Fe-rich particles is not sufficient to obtain the pure SiC sample at 1400 ^{o}C i.e. at temperatures lower than 1600 ^{o}C. This observation also supports our assumption that only the carbon obtained from novolac resin, in combination with Fe-presence, is capable of producing pure SiC at temperatures as low as 1400 ^{o}C.
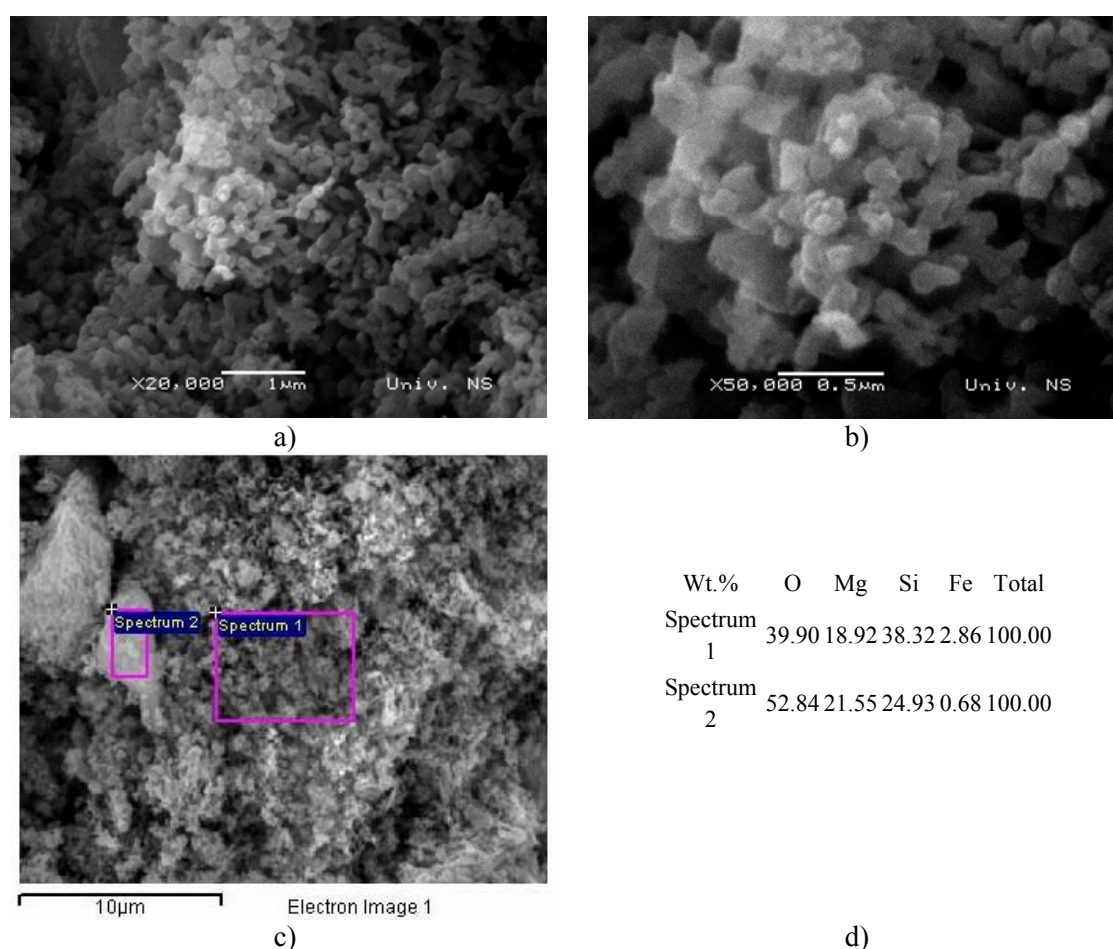

a)

b)

c)

Wt.%	O	Mg	Si	Fe	Total
Spectrum 1	39.90	18.92	38.32	2.86	100.00
Spectrum 2	52.84	21.55	24.93	0.68	100.00

d)

Fig. 10. SEM/EDS analysis of sepiolite A/saccharose sample heat treated at 1400 ^{o}C

Development of microstructure during the CTR reaction of sepiolite is illustrated in Figs. 10-12, on sepiolite A/saccharose samples heat treated in 1400-1600 ^{o}C temperature interval, together with the results of accompanying EDS analysis.

Results of SEM/EDS analysis of sepiolite A/saccharose sample heat-treated at 1400 ^{o}C are shown in Fig. 10. As one can see, its chemical composition corresponds to phase composition obtained by XRD analysis (Fig 2c): SiC, Mg$_2$SiO$_4$ and Fe$_3$Si. Spectrum 1 shows chemical composition of grey porous surface, which consists from agglomerates of very fine particles (≈100nm, Fig. 10a-b), while Spectrum 2 shows chemical composition of one of the

big particles (several μm in diameter). Content of Si and Fe is slightly higher in small particles, whilst O and Mg-content is slightly higher in big particle. However, it is obvious that SiC and forsterite phases are intimately mixed together.
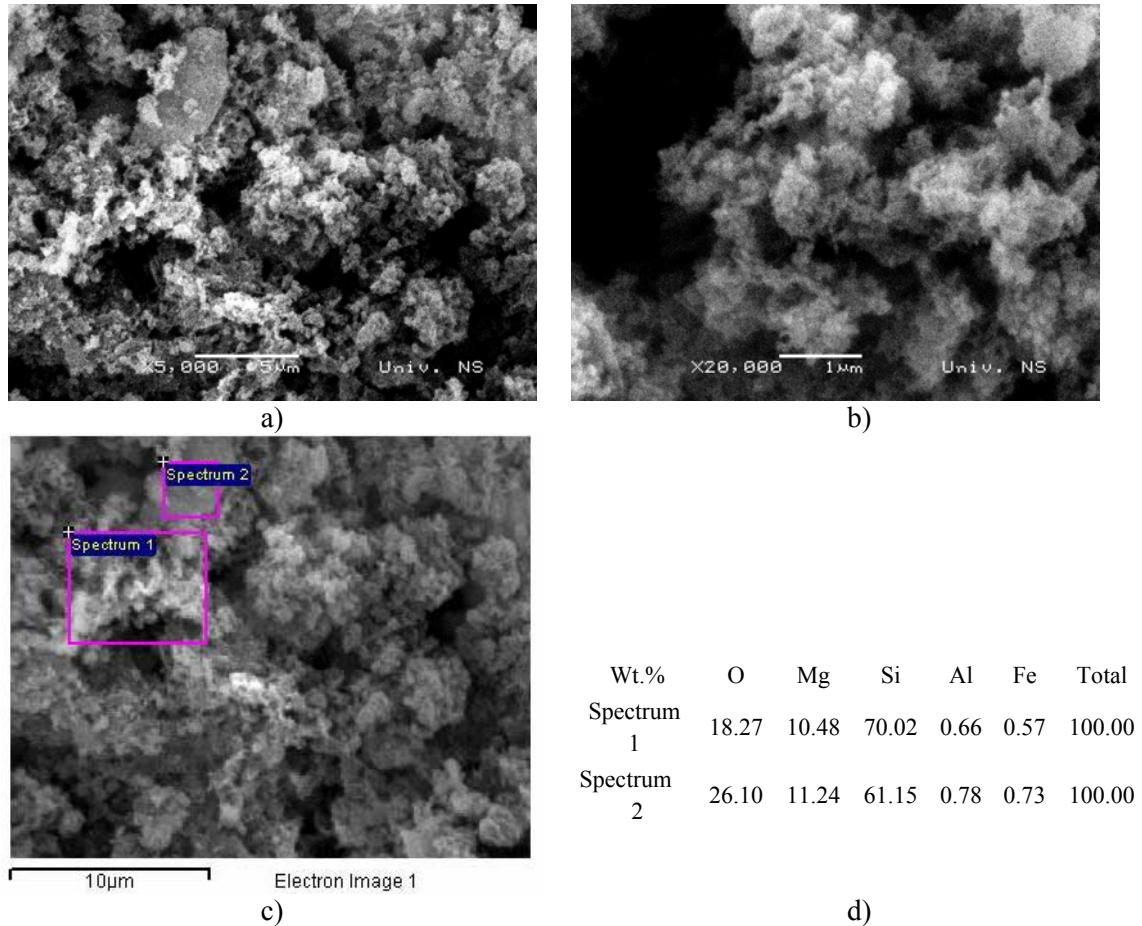

a)

b)

10μm   Electron Image 1

c)

Wt.%	O	Mg	Si	Al	Fe	Total
Spectrum 1	18.27	10.48	70.02	0.66	0.57	100.00
Spectrum 2	26.10	11.24	61.15	0.78	0.73	100.00

d)

Fig. 11. SEM/EDS analysis of sepiolite A/saccharose sample heat treated at 1500 ^{o}C

Results of SEM/EDS analysis of sepiolite A/saccharose sample heat-treated at 1500 ^{o}C are shown in Fig. 11. As one can see, its chemical composition corresponds to phase composition obtained by XRD analysis (Fig. 2d): SiC, MgO and Fe_3Si. Spectrum 1 shows chemical composition of grey porous surface, which consists from agglomerates of very fine particles (Fig. 11b), while Spectrum 2 shows chemical composition of one of the big particles (several μm in diameter). Si-content is slightly higher in small particles, whilst O-content is slightly higher in big particle. Mg, Al and Fe are distributed evenly through both small and big particles. However, it is obvious that SiC and MgO phases are intimately mixed together.

Results of SEM/EDS analysis of sepiolite A/saccharose sample heat-treated at 1600 ^{o}C are shown in Fig. 12. As one can see, its chemical composition corresponds to phase composition obtained by XRD analysis (Fig. 2e): SiC, Fe_3Si and no trace of Mg-containing compounds. Big porous particles seen in Fig. 12d, consists from agglomerates of very fine particles (≈100-200nm, Fig. 12a-b), very similarly to SiC phase observed for sepiolit A/novolac sample heat treated at 1400 ^{o}C (Fig. 8). Spectrum 1 presented in Fig. 12d represents average composition of the large area of sample. By using BSE (Back Scattering Electrons) detector mode, Fe-rich areas became clearly visible, usually as brightly white spheres (Fig. 12c). Spectrums 1 and 2 represent chemical composition of such Fe-rich areas.

On the other hand, Spectrums 3 and 4 represent chemical composition of Si-rich areas: presence of the significant amount of Fe and O indicates that these areas beside SiC, also contain some Fe_3Si phase. Impurities like Al, Cr and Ca are distributed evenly through both Fe-rich and Si-rich phase.
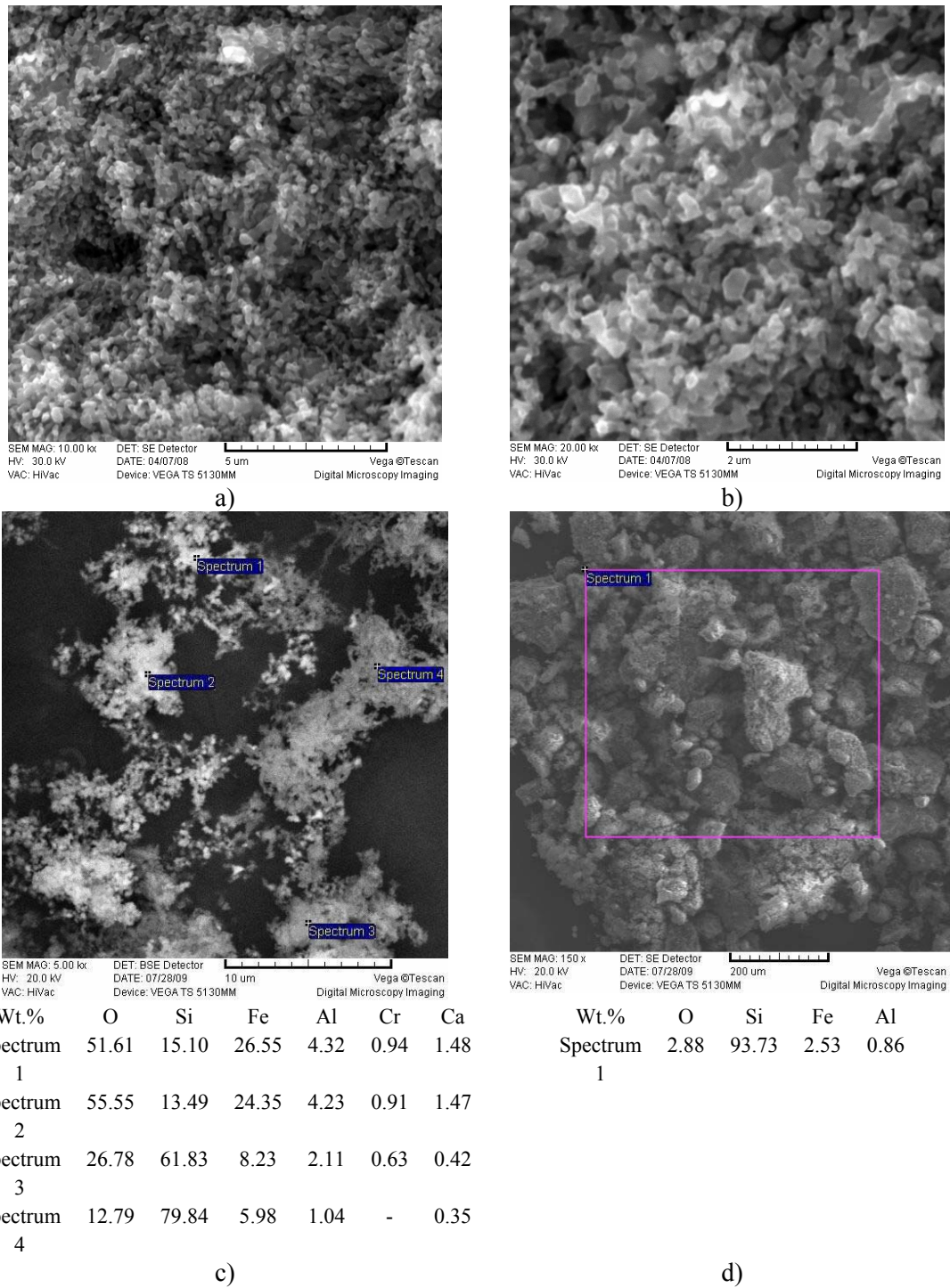

a)   b)

Wt.%	O	Si	Fe	Al	Cr	Ca
Spectrum 1	51.61	15.10	26.55	4.32	0.94	1.48
Spectrum 2	55.55	13.49	24.35	4.23	0.91	1.47
Spectrum 3	26.78	61.83	8.23	2.11	0.63	0.42
Spectrum 4	12.79	79.84	5.98	1.04	-	0.35

Wt.%	O	Si	Fe	Al
Spectrum 1	2.88	93.73	2.53	0.86

c)   d)

Fig. 12. SEM/EDS analysis of sepiolite A/saccharose sample heat treated at 1600 $^{\circ}$C

As one can see from data presented in Figs. 8-12, presence of impurities (Fe, Al, Ca, Cr) is confirmed in all sepiolite A/carbon samples. We already emphasizes that they may produce liquid phases, i.e. multiphase eutecticums and/or low temperature melting compounds (like iron-silicides), and thus have catalytic influence on the CTR reaction by facilitating the transport of the material. However, it is also obvious that their presence is not enough to obtain pure SiC powders at temperatures lower than 1600 ^{o}C.

If at all this we bear in mind that:

- in case of sepiolite B/carbon samples, including one prepared with novolac resin, pure SiC powders are obtained only at 1600 ^{o}C.

- sepiolite B/carbon samples contain much less impurities due to sepiolite B chemical composition (Tab. I), but gives pure SiC powders at the same temperature as sepiolite A/AC and sepiolite A/saccharose samples.

Then it is quite conclusive that only by combining the presence of impurities with the presence of carbon obtained from novolac resin, pure SiC powders can be obtained at temperature as low as 1400 ^{o}C. Since Fe is a major impurity in sepiolite A and that Fe_3Si is observed in XRD patterns of sepiolite A/carbon samples, it is most probably that Fe/Fe_3Si is the one responsible for catalytic influence of impurities on CTR process, probably by forming liquid phases and thus facilitating material transport and reactions (4)-(6). However, to be fully verified, these assumptions need further investigations (like TEM/EDS), and our future research will focus on catalytic influence of Fe and similar impurities (Al, Ca, Cr, etc) on CTR process, in order to clarify their true mechanism of catalytic activity.

4. Conclusions

It was shown that both chemical composition of sepiolites and type of carbon precursor used, are important factors that can significantly influence the final temperature needed for complete carbothermal reduction of sepiolites into the pure SiC powders. They are obtained at rather mild conditions(1400-1600 ^{o}C), after complete removal of the Mg-containing species. Obtained SiC powders consist from agglomerates of very fine particles ($\approx$100-200nm), i.e. obtained SiC did not retain the characteristic fibrous morphology of sepiolites. As for the influence of impurities and type of carbon used, carbon obtained from novolac resin together with the presence of Fe in the system, seems to be the most effective combination, with respect to the temperature needed for complete transformation of sepiolites into the pure SiC powders (1400 ^{o}C for sepiolite A/novolac sample vs. 1600 ^{o}C for all other samples).

Acknowledgment

5. References

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Садржај: *Силицијум карбидни (SiC) прахови су синтетисани методом карботермалне редукције на релативно ниским температурама (1200-1600°C). Као полазна сировина односно извор силицијума, коришћене су домаће минералне сировине (бели сепиолит и браон сепиолит). Установљено је да и хемијски састав сепиолита и тип коришћеног угљеника (редукционо средство), веома утичу на sat процес карботермалне редукције. Добијени силицијум карбидни прахови се састоје од финих честица бета силицијум карбида и нису задржали влакнасту морфологију полазних сировина тј. сепиолита. Опажени каталитички утицај гвожђа (Fe) је приписан формирању гвожђе силицида и њиховој улози у олакшаном уклањању магнезијумових једињења (Mg_2SiO_4 и MgO), тј. редукцији истих у гасовити магнезијум – $Mg_{(g)}$.*
Кључне речи: *Силицијум карбид; Карботермална редукција; Сепиолит; Силикати.*