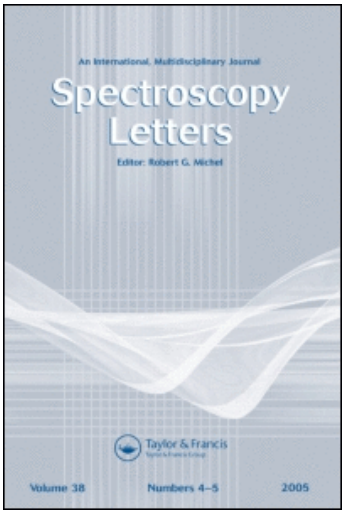

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Synthesis, Characterization, and Dissolution of Biomorphic Ceramics: Determination of Metal Ions

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ABSTRACT Converting biological structures into ceramic materials has recently received increasing interest. In the current paper, wood templates (oak and beech) with a unidirectional pore structure have been used as the carbon source. We have synthesized two new biomorphic ceramic materials from oak and beech woods and Si infiltration. After the material characterization, we have optimized the sample dissolution by acid attack in an oven under microwave irradiation. Experimental designs were used as a multivariate strategy for the evaluation of effects of varying several variables. The optimization was performed in two steps using factorial design for preliminary evaluation and a Draper–Lin design for determination of the critical experimental conditions. Five variables (time, power, volume of HNO_3, volume of H_2SO_4, and volume of HF) were considered as factors, and response was considered as the concentration of different metal ions in the optimization process.

KEYWORDS biomorphic ceramics, dissolution, experimental design, metal ions, synthesis

INTRODUCTION

Porous ceramics have received considerable interest in studies and have been used as hot gas or molten metal filters, catalyst carriers, and separation membranes because of their excellent mechanical properties, high-temperature resistance, and chemical stability.[1,2]

Design of novel ceramic structures by mimicking the cellular tissue anatomy of natural biostructures such as wood has recently become a matter of increasing interest. The highly anisotropic cellular structure of wood may be used to generate novel cellular ceramics with a micro- and mesostructure pseudomorphous to the initial porous tissue skeleton, which might be of particular interest for the applications mentioned above. Wood is a naturally grown composite material of complex hierarchical cellular structure.[3]

Biomorphic SiC is fabricated by Si infiltration of carbon templates obtained by controlled pyrolysis of wood. Through this process, the microstructure of the final SiC product mimics that of the starting wood, which has been perfected by natural evolution. The basic features of such microstructure are its

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porosity (ranging from 30% to 70%) and its anisotropy, which resembles the cellular microstructure and the mechanical characteristics of the bone.

The biomorphic ceramics[4–25] studied in this work are especially interesting because they combine the advantages of these natural structures with the capacity to be used in structural applications with high temperatures of silicon carbide.[26] Studart et al.[27] published a review about the main processing routes that can be used for the fabrication of macroporous ceramics with tailored microstructure and chemical composition.

Silicon carbide is an industrial material of great importance, with a considerable number of applications: solar cells, nuclear fuel containers, and electronic equipment. Of the different phases of silicon carbide, the β-SiC is most useful due to its high resistance to oxidation and radiation, appropriate electrical resistivity and wide hollow of bands for uses such as semiconductor, high resistance and hardness, relatively high thermal conductivity, and low thermal expansion.[28,29]

The mechanical properties and physical structures of the biomorphic materials have been studied in order to obtain information for their practical use. Generally, in the ceramic derived from wood, the mechanical properties increase with fractional density; strength and strain-to-failure in axial direction exhibit significantly higher values compared with loading in radial and tangential directions. Moreover, the elastic module and fracture toughness of biomorphic ceramics strongly depend on the properties of starting wood preforms and the degree of molten silicon infiltration.[30] On the other hand, X-ray diffraction (XRD) analysis indicated that the final product prepared by the more used technique of pyrolysis is composed of β-SiC and free silicon.[31]

The chemical composition of the biomorphic materials has been scarcely investigated. Only the presence of unreacted silicon in the structure has been evaluated by wet chemical analysis: powered samples were etched in an aqueous solution of HF/HNO_3 at room temperature, and the Si content in the solution was measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES) or inductively coupled plasma-mass spectrometry (ICP-MS).[32] However, these materials contains impurities, and co-milling of the binary compounds leads to heterogeneous distribution of impurities, which impairs the mechanical and physical properties of the final ceramic, so that the chemical analysis of these biomorphic materials is very important.

In the current work, two biomorphic materials derived from oak and beech woods are synthesized. X-ray photoelectron spectroscopy (XPS) has been used to verify the content of SiC after the preparation of the biomaterial samples. Atomic absorption spectrometry (AAS) has been used after the previous microwave dissolution of the samples; dissolutions of the samples have been optimized by using acid attack under microwave irradiation.

MATERIALS AND METHODS

Instrumentation

XPS analysis was performed with a Physical Electronics 5700 (Minneapolis, MN, USA) instrument with a MgKα X-ray excitation source ($h\nu = 1253.6$ eV); binding energies (BE) were determined with respect to the position of the Ca2p peak at 346.5 eV. The residual pressure in the analysis chamber was maintained below 10^{-9} Torr during data acquisition.

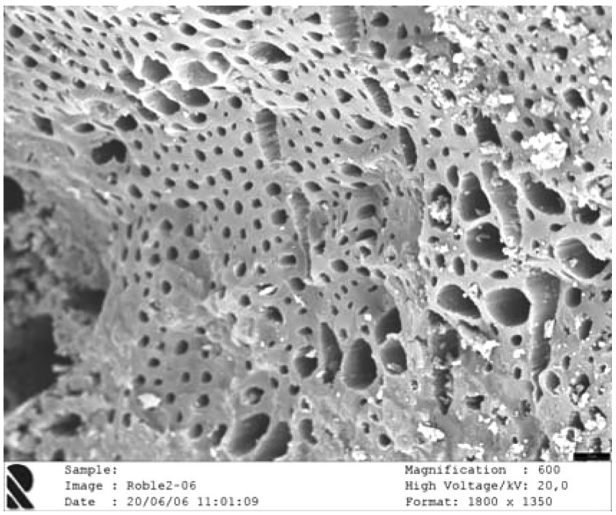
Scanning electronic microscopy (SEM; JEOL, model 840, Tokyo, Japan) was used to obtain the micrographs shown in Figs. 1 and 2.

A Panasonic (National) microwave oven (model NN-8507, Trenton, NJ, USA) and a Parr Microwave Acid Digestion Bomb (model 4782, Boston, MA, USA) were used for sample digestion. The bombs were cleaned before use with 10% nitric acid for 1 day followed by repeated rinsing with water.

A Varian model SpectrAA 50 (Mulgrave, Victoria, Australia) flame atomic absorption spectrometer was used for the analysis with the appropriate hollow cathode lamp.

Reagents

Analytical reagent grade chemicals were used throughout. Standard 1000 μg mL^{-1} Fe(III), Al(III), and Zn(II) solutions (Fluka, Buchs SG, Switzerland) were used. Standards of working strength were made by appropriate dilution as required, immediately prior to use. Water was deionized with a Milli-Q system (Boston, MA, USA). Concentrated acid HF, H_2SO_4, and HNO_3 (Merck, Damstadt, Germany) were used for digestion of the samples.



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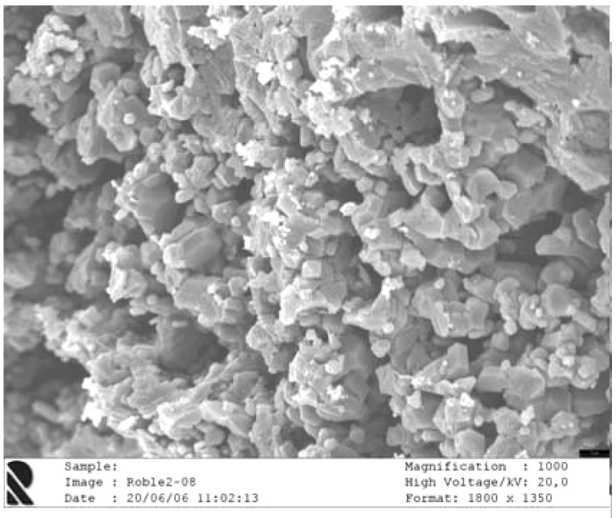

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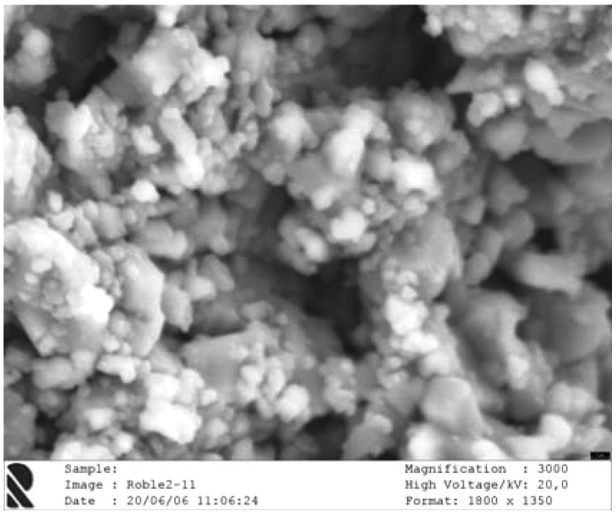

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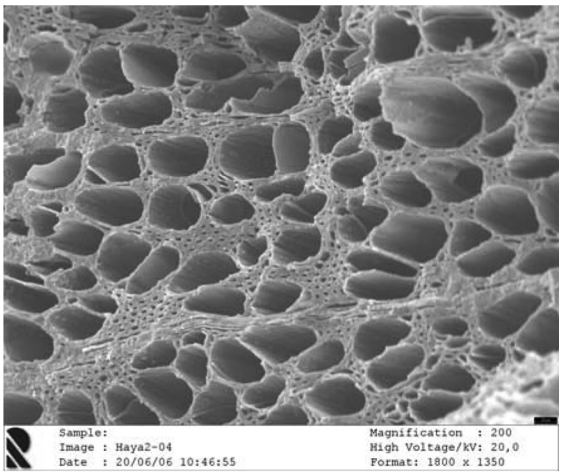
FIGURE 1 **Micrographs of oak biomorphic material obtained with SEM at different magnification.**



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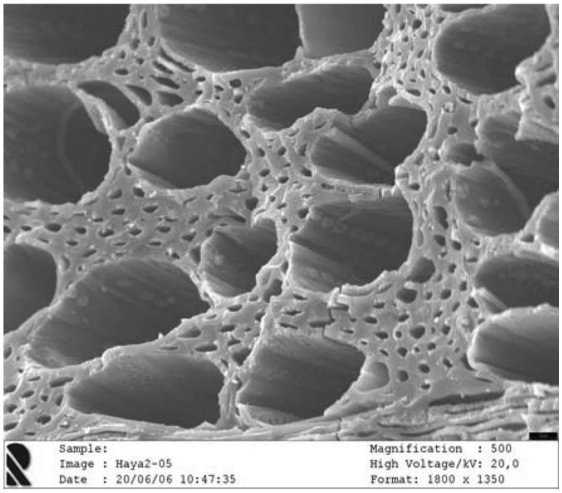

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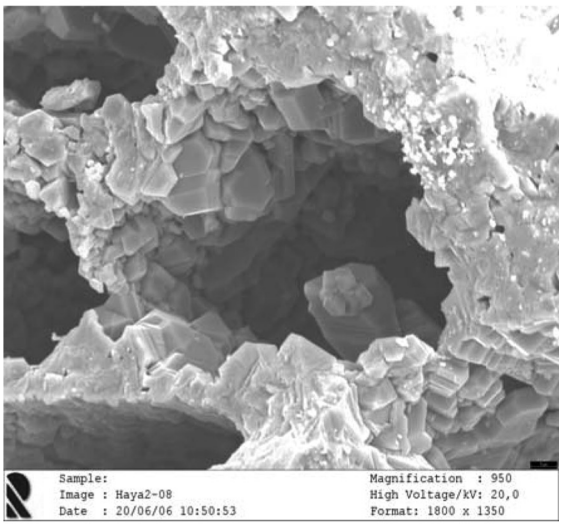

(c)

FIGURE 2 **Micrographs of beech biomorphic material obtained with SEM at different magnification.**

TABLE 1 Factor Levels in the Design

Variable	Lower level (−)	Middle level (0)	Upper level (+)
Volume of HF (mL)	1	2.5	4
Volume of HNO_3 (mL)	1	2.5	4
Volume of H_2SO_4 (mL)	1	2.5	4
Power (W)	400	625	850
Time (min)	1	2.5	4

Synthesis

Oak and beech woods were dried to 70°C during 15 h, and the pyrolysis was divided in two stages, both in atmosphere of Ar:

- Slow heating to 1°C/min until reaching a temperature of 500°C; in this stage, the polyaromatic hydrocarbon polymers (cellulose, hemicellulose, and lignin) are disturbed completely and form carbon.
- Heating at greater speed (5°C/min) until reaching the final temperature of 1500°C with a dwell time in the same one of 4 h.

Vacuum Si infiltration: For oak wood with Si powder in a molar ratio C:Si of 1:1, heating was at 20°C/min until 1600°C for 4 h. For beech wood, Si pieces were used in a molar ratio of 1:1.5, and heating was at 15°C/min until 1600°C for 4 h.

Optimization Strategy

The experimental variables were optimized by applying an exploratory fractional factorial experimental design at three levels, involving 14 runs that were used as the first approximation to the Draper–Lin small-composite response surface design which was used to optimize the digestion process. Table 1 lists the upper, middle, and lower values given to each factor, and Table 2 shows the experimental design matrix. The experimental data were processed making use of the STATGRAPHICS 5.1 plus program.[33] The significance of the effects was done by analysis of variance (ANOVA) and using p value significance levels. This value represents the probability of the effect of a factor being due solely

TABLE 2 Draper–Lin Design Matrix and the Results as Average Fe, Al, and Zn Concentrations

No.	Volume HF (mL)	Volume HNO_3 (mL)	Volume H_2SO_4 (mL)	Power (W)	Time (min)	Fe Oak	Fe Beech	Al Oak	Al Beech	Zn Oak	Zn Beech
						($\mu g\ mL^{-1}$)					
1	2.5	2.5	4.0	625	2.5	0.24	0.23	31.25	26.02	0.07	0.04
2	1.0	2.5	2.5	625	2.5	0.19	0.09	29.00	30.31	0.14	0.06
3	2.5	1.0	2.5	625	2.5	0.14	0.16	38.00	7.17	0.12	0.04
4	2.5	2.5	2.5	625	4.0	0.18	0.12	31.75	5.83	0.06	0.05
5	2.5	4.0	2.5	625	2.5	0.13	0.10	33.20	2.17	0.09	0.04
6	4.0	4.0	4.0	400	1.0	0.06	0.21	28.33	8.08	0.06	0.04
7	1.0	1.0	1.0	850	1.0	0.1	0.06	35.13	21.75	0.06	0.03
8	2.5	2.5	2.5	625	1.0	0.09	0.15	35.56	16.02	0.05	0.04
9	4.0	1.0	4.0	850	1.0	0.20	0.15	34.77	18.15	0.06	0.03
10	1.0	1.0	1.0	400	1.0	0.07	0.06	45.17	21.11	0.04	0.03
11	4.0	4.0	1.0	400	1.0	0.09	0.05	41.16	16.95	0.07	0.03
12	2.5	2.5	1.0	625	2.5	0.10	0.05	46.98	20.87	0.07	0.03
13	2.5	2.5	2.5	400	2.5	0.12	0.06	30.09	21.46	0.04	0.04
14	4.0	1.0	1.0	400	4.0	0.09	0.08	28.36	20.87	0.05	0.03
15	2.5	2.5	2.5	625	2.5	0.14	0.12	42.94	20.72	0.05	0.02
16	4.0	4.0	1.0	850	4.0	0.13	0.11	15.20	14.00	0.01	0.02
17	1.0	1.0	4.0	400	4.0	0.24	0.15	23.00	15.50	0.05	0.05
18	2.5	2.5	2.5	625	2.5	0.11	0.09	12.46	11.80	0.04	0.04
19	4.0	1.0	4.0	850	4.0	0.23	0.19	14.50	17.47	0.10	0.05
20	4.0	2.5	2.5	625	2.5	0.12	0.12	12.50	18.50	0.12	0.03
21	1.0	4.0	4.0	850	4.0	0.14	0.08	21.29	10.67	0.07	0.03
22	1.0	4.0	1.0	850	1.0	0.1	0.06	42.50	19.98	0.06	0.02
23	2.5	2.5	2.5	850	2.5	0.1	0.09	44.02	7.87	0.10	0.02
24	1.0	4.0	4.0	400	4.0	0.2	0.13	34.84	10.00	0.09	0.03

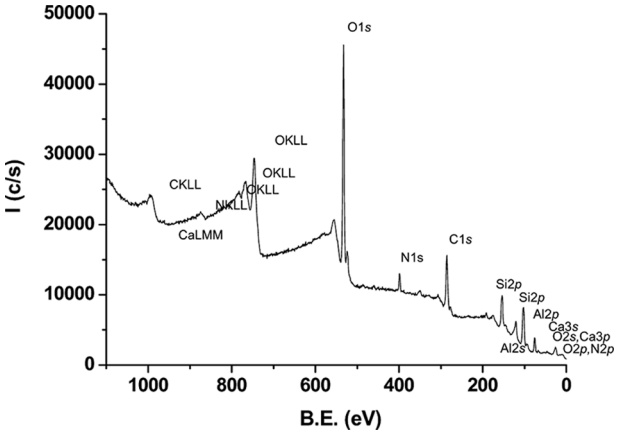

FIGURE 3 XPS spectrum of biomorphic material from oak wood.

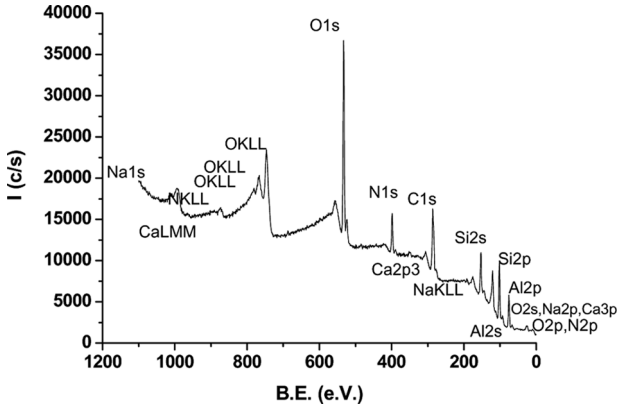

FIGURE 4 XPS spectrum of biomorphic material from beech wood.

to random error. Thus, if the p value is less than 5%, the effect of corresponding factor is significant.

RESULTS AND DISCUSSION

XPS Analysis

XPS has been used for the study of the surface composition (major and minor) of the biomorphic samples. XPS spectra obtained are shown in Figs. 3 and 4, and deconvolutions of the C1s and Si2p peaks has been carried out and are shown in Figs. 5 and 6, respectively. Consequently, the performance of the synthesis of biomorphic ceramics can be evaluated approximately from these data. The relative atomic concentrations obtained expressed as a percentage are as follows: Oak wood, C*1s* 26.55; Si*2p* 20.25; O*1s* 40.97; N*1s* 4.67; Al*2p* 7.21; Ca*2p* 0.36. Beech wood, C*1s* 30.12; Si*2p* 17.41; O*1s* 28.79; N*1s* 8.47; Al*2p* 14.63; Ca*2p* 0.30.

Sample Dissolution

The principal problem in the determination of metal species present in the biomorphic ceramic is the difficult dissolution of the samples due to the high resistance of these advanced ceramics to thermal and chemical attack. Although decomposition by alkali fusion can be used, this procedure is slow and implies the use of a platinum crucible,[34] and the presence of an excess of solid fusion reagent. For this reason, in the current work dissolution has been achieved by acid attack ($HF + HNO_3 + H_2SO_4$) under microwave-assisted pressure digestion.

The advantages of microwave dissolution include faster reaction rates that result from the high temperature and pressures attained inside the sealed containers. The use of closed vessels makes it possible to eliminate uncontrolled trace element losses of volatile

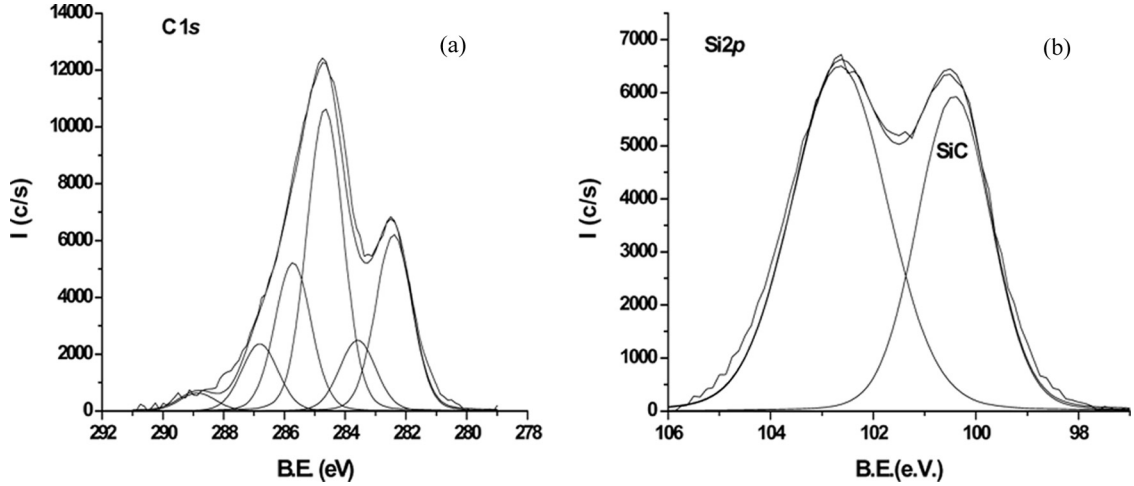

FIGURE 5 Deconvolution bands from XPS spectrum from oak wood: (a) C*1s*; (b) Si*2p*.

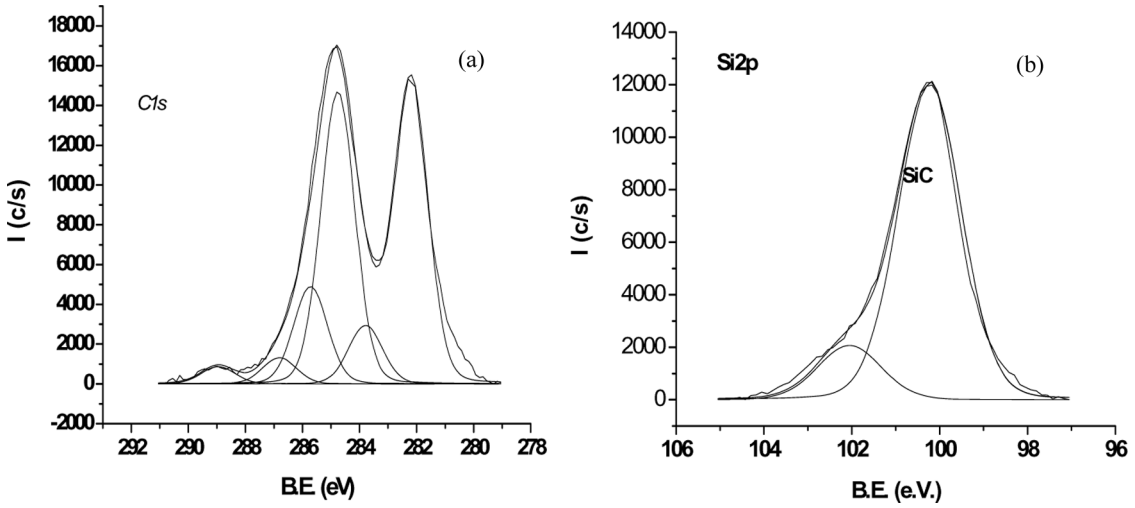

FIGURE 6 **Deconvolution bands from XPS spectrum from beech wood: (a) C*1s*; (b) Si*2p*.**

molecular species that are present in a sample. Another advantage of microwave dissolution is a decrease in blank values compared with open-beaker work, both because contamination from the laboratory environment is much lower and because closed vessels make it possible to use smaller quantities of reagents.

Different times and power settings were investigated to establish the proper settings required for the digestion of biomorphic ceramic samples without damage to the digestion vessels. Maximum load of the bomb heating at 100% power at the start of the dissolution caused leakage and deformation of the

TABLE 3 **p Value for Data Analysis Given in Table 2 for Metal Ions Concentrations**

Source	Fe Oak	Fe Beech	Al Oak	Al Beech	Zn Oak	Zn Beech
			p Value			
A: Power	0.3430	0.9037	0.5946	0.0751	0.2887	0.3671
B: Time	0.0267	0.9037	0.9855	0.3398	0.7820	0.3445
C: Vol H_2SO_4	0.0111	0.0266	0.3560	0.767	0.9445	0.5991
D: Vol HF	0.0758	0.1114	0.6713	0.2836	0.5043	0.8034
E: Vol HNO_3	0.5331	0.2438	0.5133	0.2582	0.7304	0.1556
AA	0.0783	0.1078	0.7198	0.8005	0.4297	0.1738
AB	0.4799	0.1203	0.6767	0.6823	0.4421	0.5593
AC	0.0724	0.327	0.6206	0.5111	0.7953	0.4212
AD	0.1815	0.7576	0.7530	0.4141	0.6410	0.2676
AE	0.1060	0.3138	0.7430	0.3115	0.9388	0.7739
BB	0.6481	0.4286	0.9963	0.2341	0.2085	0.5109
BC	0.3951	0.8661	0.8261	0.4569	0.7953	0.2964
BD	0.1420	0.2892	0.8936	0.3508	0.6619	0.5422
BE	0.0763	0.9929	0.2665	0.0955	0.3455	0.3768
CC	0.0901	0.3237	0.5735	0.0829	0.4297	0.5483
CD	0.4500	0.7186	0.4522	0.0673	0.3455	0.3413
CE	0.0405	0.2951	0.8306	0.7669	0.5434	0.3971
DD	0.3180	0.5634	0.8346	0.0392	0.5385	0.7649
DE	0.1001	0.88	0.9586	0.3396	0.7722	0.2906
EE	0.6481	0.5459	0.2375	0.0638	0.1635	0.3550
			R-squared (%)			
	98.4730	95.5816	77.6800	93.523	83.272	86.8432

TABLE 4 Optimum Values of the Factors for Metal Ions Determination

	Fe		Al		Zn	
Factor	Oak	Beech	Oak	Beech	Oak	Beech
Power	808	448	850	465	805	850
Time	4.0	1.0	4.0	1.0	4.0	4.0
Volume of H_2SO_4	4.0	4.0	1.3	4.0	2.2	1.0
Volume of HF	1.0	2.2	1.0	1.2	1.0	1.0
Volume of HNO_3	1.4	1.0	2.1	1.5	1.1	2.4

vessels. Lower power settings permit controlled decompositions.

The content of metal species in the samples has been evaluated by AAS. The standard addition procedure has been used in all cases. For the digestion and determination, 10 mg of powdered biomorphic samples were weighed exactly, added to the digestion vessel with the acid mixture, and digested in the microwave oven using the conditions listed in Table 2. After digestion, the samples were diluted to 25 mL with deionized water in a calibrated flask. For the analysis by standard additions of these digested samples, a blank solution prepared with the same volumes of acids as the samples raised to 25 mL and three sample solutions with standard additions of the elements were used to obtain the calibration graphs.

The results of the ANOVA carried out on the data given in Table 2 are shown in Table 3. The optimum values for the determination of each metal ion are shown in Table 4.

CONCLUSIONS

XPS is the more useful device to obtain fast information about the content of silicon carbide in the samples; this fact is very interesting as a tool to verify the results of the synthesis of these materials.

The determination of metal ions in these ceramics can be achieved by acids dissolution under microwave irradiation, followed by AAS analysis, although the solubilization step is time-consuming and the use of high-purity acids is absolutely necessary. Dissolution under microwave irradiation shortens appreciably the time of the acid attack.

Optimization of digestion procedures, based on a traditional univariate approach, involves many experiments. In this study, it has been shown that the application of a Draper–Lin design considerably reduces the amount of experimental work required. By using such a design, we have been able to establish the optimum conditions for the determination of Fe(III), Al(III), and Zn(II). Furthermore, as atomic spectroscopy moves more toward multivariate analysis, the need for multivariate experimental design and optimization techniques becomes important in establishing valid methodology.

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