

Effect of Forming Methods on the Properties of Controlled Porous Ceramics

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Abstract

Two methods of forming, namely, semi-dry press and slip casting were used to fabricate porous ceramics from local raw materials, kaolin and silica. Different wt.% of aluminum-zinc mixture powders were used as gas creative agent in chemical swelling method to create porosity in ceramic slurry before forming. Dried formed ceramics were fired at four different firing temperatures of (900, 1000, 1100 and 1200)°C.

It was found that the forming technique has a great effect on the properties of the fabricated porous ceramics. From porosity point of view, slip casting is better technique to produce high porous ceramics, but semi-dry pressing seems to be good for other physical properties and mechanical properties.

الخلاصة:

استخدمت طريقتان لتشكيل المواد السيراميكية وبالتحديد طريقتي الكبس شبه الجاف والصب الانزلاقي لتصنيع سيراميك مسامي من بعض المواد الخام المحلية كالكاؤولين والسيليكا. استعمل مزيج من مسحوق الألمنيوم والزنك لتوليد الغازات خلال عملية التحضير بطريقة الانتفاخ الكيماوي لتوليد المسامية في الخلطة السيراميكية قبل عملية التشكيل. حرقت النماذج المشكلة بأربع درجات حرارية (٩٠٠، ١٠٠٠، ١١٠٠ و ١٢٠٠)°م. لقد وجد أن طريقة التشكيل المستخدمة في التصنيع لها تأثير كبير في خصائص الأجسام السيراميكية الناتجة. فمن ناحية المسامية وجد بأن التشكيل بالصب الانزلاقي يعطي أجسام ذات مسامية أعلى من طريقة التشكيل بالكبس شبه الجاف الذي امتازت سيراميكياته المصنعة بجودة الخصائص الفيزيائية والميكانيكية.

Introduction:

The tremendous strides made the last two decades in the advancement of technology related to a requirement for quite a large number of parts with controlled porosity. In ceramic technology, obtaining a porous product of such parameters and simultaneously showing suitable mechanical strength is not an easy matter [1,2].

In our current attempts on fabrication of controlled porous ceramics using organic materials [3] or non-ferrous metal [4], constituents are treated to evolve a gas which creates bubbles in the ceramic body or organic material respectively which leads to fluffing of the mass, and ensure

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high porosity of the ceramic materials after firing process

Due to both, the suitable selection of grain composition of basic materials, the kind and wt.% of the additives helping to emit the gas, as well as the change in viscosity of the initial suspension, it is possible to regulate quantity and size distribution of origination pores to a great extent, especially when chemical swelling methods, are used [5].

In all our previous works, uniaxial semi-dry pressing technique was used to form a ceramic body under different loads, and 3 tons, corresponding to 61.8 MPa, was found to produce the best properties [3]

. The excellent properties obtained are related to different reasons, one of which is the applied load used in forming method increases the compaction of all mixes ingredients, and with the aid of high firing temperature, the densification is increased and this is positively reflected on the strength of fabricated ceramics, although high level of porosity is obtained.

In this work, another forming technique, namely slip-casting, where no load needs to be used, is employed to fabricate ceramics and compared their Properties are with those of pressed ceramics processed under the same conditions.

Experimental:

1- Raw Materials

Kaolin and silica ceramic raw materials, from western desert of Iraq, were used in this work. They were sieved through 63 μm screen.

2- Additives.

Different wt.% mixtures of aluminum powder and zinc powder, both having 10 μm particle size, were used as gas creative agents in ceramics due to their reaction with dilute sodium hydroxide solution in a molar ratio [6,7].

3- Sample Prepration.

Samples were made from kaolin and silica having no additives and with different wt.% of additives as shown below

a) Semi-Dry Pressing Method.

Uniaxial semi-dry pressing technique was employed for forming ceramic samples for batches prepared in proportions as shown in Table (1). All samples were pressed in cylindrical form by using carbon steel mould, and they had the diameter of 25 mm and 5 mm in height (thickness).

(b) Slip-Casting Method.

Slip-casting was is used for forming samples for batches prepared in proportions as shown in Table (2). The slip is poured into a plaster of Paris mould to produce ceramic in cylindrical shape. The mould was left until the cast dries. Then the mould is left for 24 hrs before another casting.

4- Drying And Firing.

In each of the processes, which requires the addition of some water content, the drying step in which the liquid has be removed, must be carefully controlled for satisfactory results. Samples were dried first for 48 hrs at temperature and then on an oven for 24 hrs at 110°C.

Firing of all the samples was carried out at 900, 1000, 1100 and 1200°C in a carbolite furnace. All samples were soaked for one hour at each firing temperature.

Results And Discussion:

It is known that the shaped ceramic articles suffer from shrinkage during drying and firing processes, and the shrinkage takes place as a result of materials being transported by one or more of several diffusion process [8,9]. This shrinkage simply appears in their volume. Linear shrinkage of fabricated ceramic was calculated by using a micrometer to measure the diameter or thickness of specimen [10]. The results obtained for linear shrinkage with no additive are plotted in Fig. (1).

It is shown that the linear % L.Sh. increases with increase in firing temperature. This may be attributed to the effect of high temperature on the volume of material which decreases the linear shrinkage due to the movement of the material particles into the pore spaces, leading to densification and decreasing the porosity of the ceramic body [11], i.e. increasing linear shrinkage.

The linear shrinkage of ceramics which were made by slip-casting as shown in Fig. (2) is greater than of the

Ceramics pressed under 3 tons. The higher the moulding pressure the lower in burning shrinkage [12].

Ceramics which are additive in their origin mixes, i.e. mixture of Al:Zn powders, exhibit different behaviours. The results obtained can be seen in Fig. (2a) for pressed ceramics. It is quite obvious that the % L.Sh. at first It is known that the shaped ceramic articles suffering from shrinkage during drying and firing processes, and the shrinkage take place as a result of materials being transported by one or more of several diffusion process [8,9]. This shrinkage is simply appeared in their volume. Linear shrinkage of fabricated ceramic was calculated by using a micrometer to measure the diameter or thickness of specimen [10]. The results obtained for linear shrinkage with no additive are plotted in Fig. (1).

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The linear shrinkage of ceramics which were made by slip-casting as shown in Fig. (2) is greater than of the ceramics pressed under 3 tons. The higher the moulding pressure the lower in burning shrinkage [12].

Ceramics had an additives in their origin mixes, i.e. mixture of Al:Zn powders, exhibited different behaviours. The results obtained can be seen in Fig. (2a) for pressed ceramics. It is quite obvious that the % L.Sh. at first two firing temperature (900 and 1000 °C) decreases with the increase wt.% addition of Al:Zn powders, Fig (2a), where at 1100 °C the behaviour seems to be different, at low wt.% addition, the linear shrinkage decreases

two firing temperatures (900 and 1000 °C) decreases with the increase in wt.% addition of Al:Zn powders, Fig (2a), where at 1100 °C the behaviour seems to be different, at low wt.% addition, the linear shrinkage decreases then increases at addition above 5 wt.%, while linear shrinkage at 1200 °C with all wt.% of Al:Zn powders. The above behaviour was directly attributed to the formation of new phases, generated from increasing concentration of zinc powder in each addition, namely Gahnite ($ZnAl_2O_4$) and Willemnite ($ZnSiO_4$) and the later is known to form a crystalline glass [13] as detected by X-ray diffraction spectroscopy [4,7].

In slip-casting ceramics, it was found that the L.Sh. decrease with increase of Al:Zn powder addition at first three temperatures (900, 1000 and 1100) °C it was slightly decreases and then increases sharply at addition above 7 wt% .

The above behaviour of linear shrinkage in ceramic having addition in their mixes or without addition was slightly directly reflected on the measurement of apparent solid density (A.S.D.) which was measured by the standard test of ASTM (C20-83). The results obtained are shown in Fig. (3) for pressed and slip-caste ceramics had no additives and in Fig. (4) for ceramics produced by both methods having different wt.% of Al:Zn in their origin mixes.

The apparent solid density was found to increase with firing temperature rise. This is due to enhancement of sintering which causes better densification and reduction of volume [14,15], Fig. (3). Addition of different wt.% of Al:Zn powders and due to their reactions with sodium hydroxide and liberation of hydrogen gas which creates porosity in the origin mixes and accordingly decreasing the

apparent solid density at low firing temperature. This effect can be seen very clearly in Fig. (4a) for pressed ceramic and in Fig. (4b) for slip-casting. Firing at higher different temperature improves the density and this improvement is also related to the amount of additives. Again pressed ceramics produces high A.S.D. than slip-casting ceramics in which no pressure (load) was used.

A knowledge of the porosity of a ceramic body is of importance for it service as a specific maturing and also allows the evaluation of this body for a specific purpose [16]. In this work, apparent porosity was measured using Helium Porosimeter model 3020-060 at Oil Exploration Company. The results obtained for ceramics with no additives for both pressed and slip-casting ceramics are plotted in Fig. (5) and those with additives in Fig. (6).

As expected, ceramics prepared by slip-casting method of forming as a technique resulted in higher porous ceramics than those prepared using pressing technique, for both cases with or without additives. It is worth to mention here that the same behaviour was observed with increasing wt.% addition as in case of linear shrinkage and apparent solid density measurements.

The diametrical compression test using Brazilian Fracture method [17,18] on an Instron 1195 and impact strength using Izod method [18] were carried out for all fired samples having no additives, Fig. (7). The results obtained for ceramics had additives in their mixes are exhibited in Fig. (8a) and Fig. (9a) for pressed samples and at Fig. (8b) and Fig. (9b) for slip-casting samples respectively. It was found that good strength, in compression and impact, was obtained with ceramics fabricated by slip-casting technique, but they are all less

than that of pressed ceramics because compression and impact, was obtained with ceramics fabricated by slip-casting technique, but they are all less than that of pressed ceramics because of its high porosity in addition to no load was used during their processing. This was also confirmed by scanning electron microscope, SEM-Jeol-JFC-1100E ion sputter, Fig. (10).

Conclusions:

1. The possibility of manufacturing porous ceramics from Iraqi kaolin and silica using different forming techniques, i.e. semi-dry and slip-casting, is achieved.
2. The forming method used is the main factor affecting the quantity and the proper and the firing temperature. porous ceramics, besides the chemical composition and the firing temperature.
3. In slip-casting, ceramics porosity is higher than in semi-dry press, while the other physical properties and the mechanical properties are lower.

References:

- [1] Rother G.R., Powder Metallurgy, **No.2,85** (1978).
- [2] Al-Lami H.S. and Adei, A.J. Eng. & Tech., **Vol.19, No.4**, 408 (2000).
- [3] Al-Lami H.S. and Sakhria Sh.A., Eng. & Tech., **Vol.21, No.3**, 176 (2002).
- [4] Al-Lami H.S., Al-Rawi W.A. and . Saadallah M.A, Eng. & Tech. **Vol.21, No.6**, 703 (2002).
- [5] Al-Lami H.S. and Sakhria, Sh.A. Iraqi Patent, **2966** (2001).
- [6] Solsarczyk, A. Zak H. and A. Towarnica, Interceram, **No.2**, 35 (1985).
- [7] Al-Rawi W.A., "Using Non-Ferrous Metals in Processing of Porous Ceramics", M.Sc. Thesis, University of Technology (1999).
- [8] Kingry H.K., Bowen H.K. and Uhlmann D.R., "Introduction to Ceramics", 2nd Ed. John-Wiley & Sons, New York (1976).

- [9] Onoda G.Y. and Hench L.L., “Ceramic Processing before Firing”, John-Wiley & Sons, New York (1978).
- [10] William R., “Properties of Ceramic Raw Materials”, 2nd Ed., Pergamon Press, UK (1978).
- [11] Budnikor, P.P. “The Technology of Ceramics and Refractories”, MIT Press, USA (1964).
- [12] Norton F.H., “Refractories”, 4th Ed., MacGraw Hill, New York (1968).
- [13] Singer F. and Singer S., “Industrial Ceramics”, Chapman and Hall Ltd., London (1963).
- [14] Looms T.S., “Insulators for High Voltages”, Peter Peregrines Ltd., London (1988).
- [15] صالح أمين، ولبد محمد، طالب الشريف،
"الخواص المواد الهندسية"، دار الكتاب-بغداد
- [16] Scheldegger A.E., “The Physical of Flow through Porous Media”, 3rd Ed., University of Toronto Press, Canada (1972).
- [17] Cahn R.W., Thompson M.W. and World, I.M. “Mechanical Behaviour of Ceramics”, Alden Press, Oxford (1980).
- [18] Al-Lami H.S. and Al-Obaidi, I.M. Iraqi Patent, **2984** (2001).

Table (1): Conditions of fabrication ceramics samples using semi-dry pressing.

Mix No.	Kaolin		Silica		Al:Zn (wt.%)	Forming load (ton)	Firing Temperature (°C)
	(wt.%)	Particle size (µm)	(wt.%)	Particle size (µm)			
Cp ₀	70	63	30	63	—	3	900,1000,1100,1200
Cp ₁	69	63	29	63	2	3	900,1000,1100,1200
Cp ₂	67.5	63	27.5	63	5	3	900,1000,1100,1200
Cp ₃	66.5	63	26.5	63	7	3	900,1000,1100,1200
Cp ₄	65	63	25	63	10	3	900,1000,1100,1200
Cp ₅	64	63	24	63	12	3	900,1000,1100,1200
Cp ₆	62.5	63	22.5	63	15	3	900,1000,1100,1200

Table (2): Conditions of fabrication ceramics samples using slip-casting.

Mix No.	Kaolin		Silica		Al:Zn (wt.%)	Firing Temperature (°C)
	(wt.%)	Particle size (µm)	(wt.%)	Particle size (µm)		
Cs ₀	70	63	30	63	—	900,1000,1100,1200
Cs ₁	69	63	29	63	2	900,1000,1100,1200
Cs ₂	67.5	63	27.5	63	5	900,1000,1100,1200
Cs ₃	66.5	63	26.5	63	7	900,1000,1100,1200
Cs ₄	65	63	25	63	10	900,1000,1100,1200
Cs ₅	64	63	24	63	12	900,1000,1100,1200
Cs ₆	62.5	63	22.5	63	15	900,1000,1100,1200

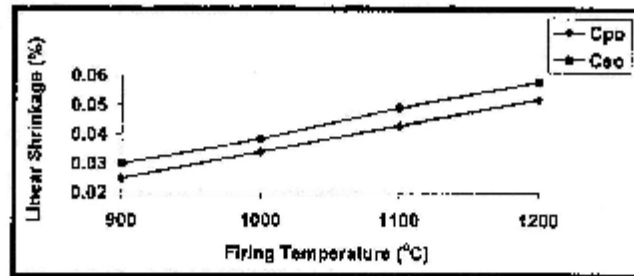


Fig. (1) Effect of firing temperature on the linear shrinkage of prepared ceramics having no additive (Cp semi-dry pressing, Cs slip-casting).

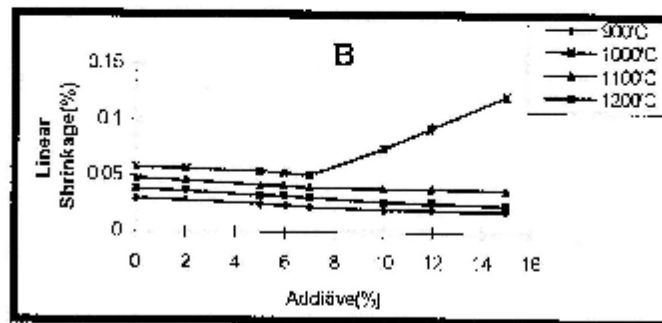
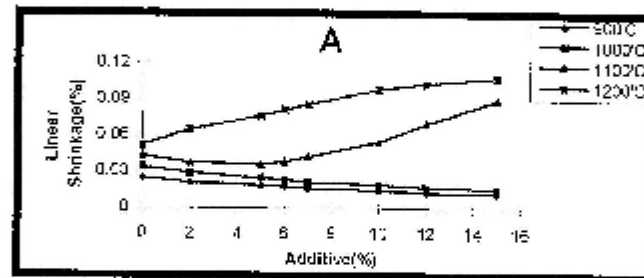


Fig (2) Effect of wt% addition on the linear shrinkage of ceramics fired different temperature and formed by (a) semi-dry pressing, and (b) slip casting.

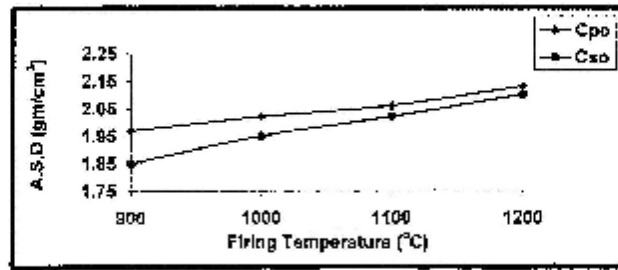


Fig. (3) Effect of firing temperature on the apparent solid density of prepared ceramics having no additive (Cp semi-dry pressing, Cs slip-casting).

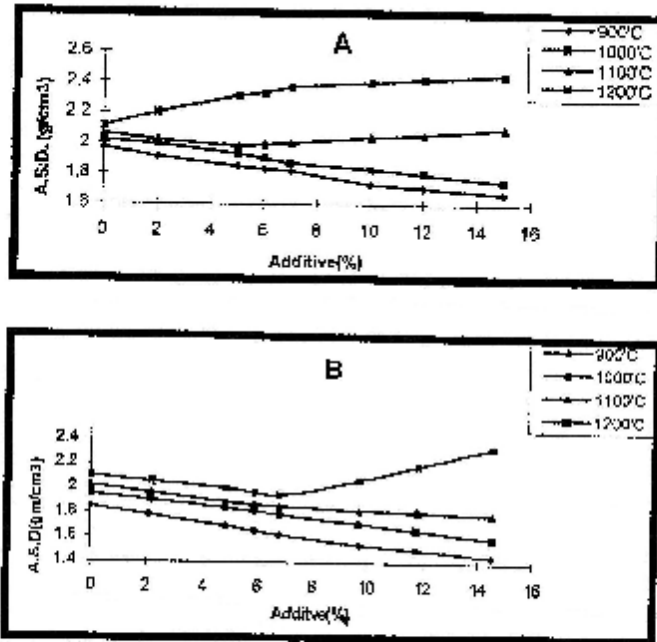


Fig. (4) Effect of wt% additive on apparent solid density of fabricated ceramic fired at different temperature and formed by (A) semi-dry Pressing, (B) slip-casting.

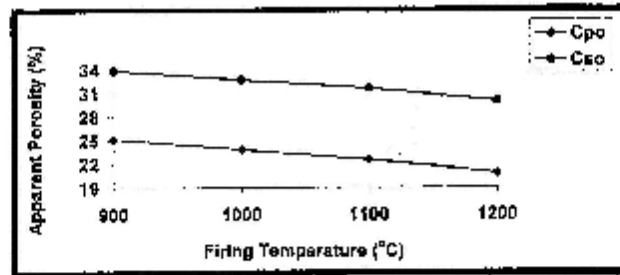


Fig. (5) Effect of firing temperature on the apparent porosity of The ceramic having no additive (Cp semi-dry pressing, Cs slip-casting).

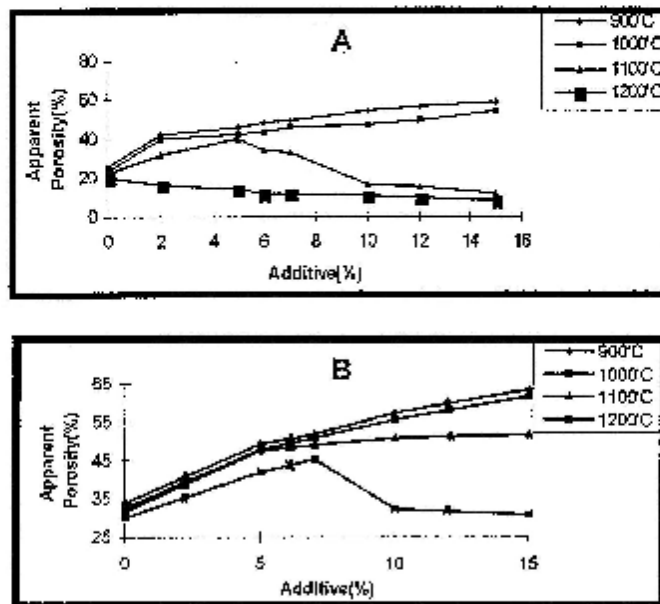


Fig. (6) Effect of wt% on apparent porosity of the resulted ceramics Fired at different temperature and formed by (A) semi-dry pressing (B) slip casting.

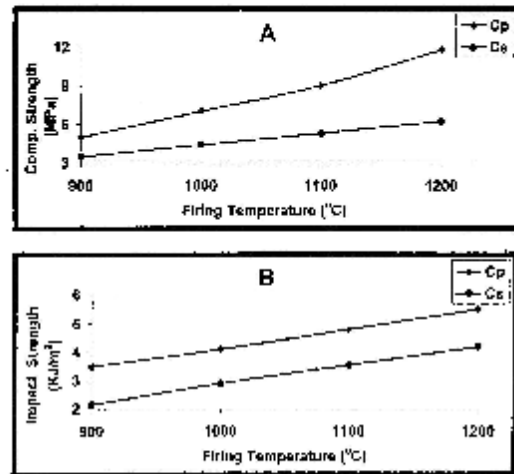


Fig. (7) Effect of firing temperature on the (A) compressive strength and (B) impact strength of the ceramics having no additive (Cp semi-dry pressing, Cs slip-casting).

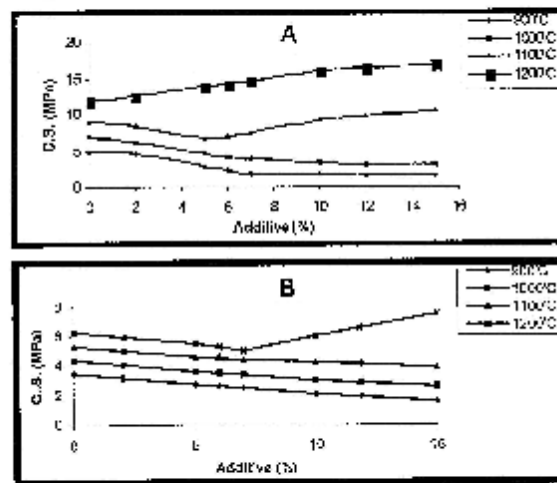


Fig. (8) Effect of wt% on compressive strength of fabricated ceramics fired at different temperature and formed by (A) semi-dry pressing, (B) slip-casting.

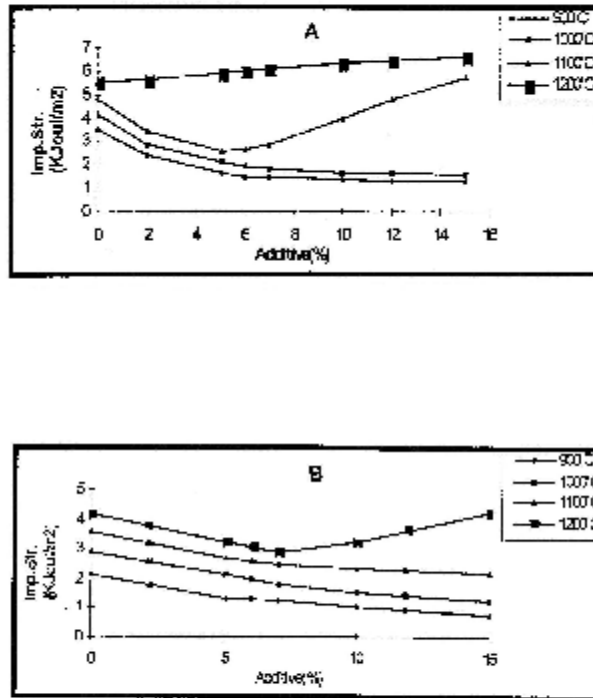


Fig. (9) Effect of wt% on Impact strength of fabricated ceramics fired at different temperature and formed by (A) semi-dry pressing, (B) slip-casting.

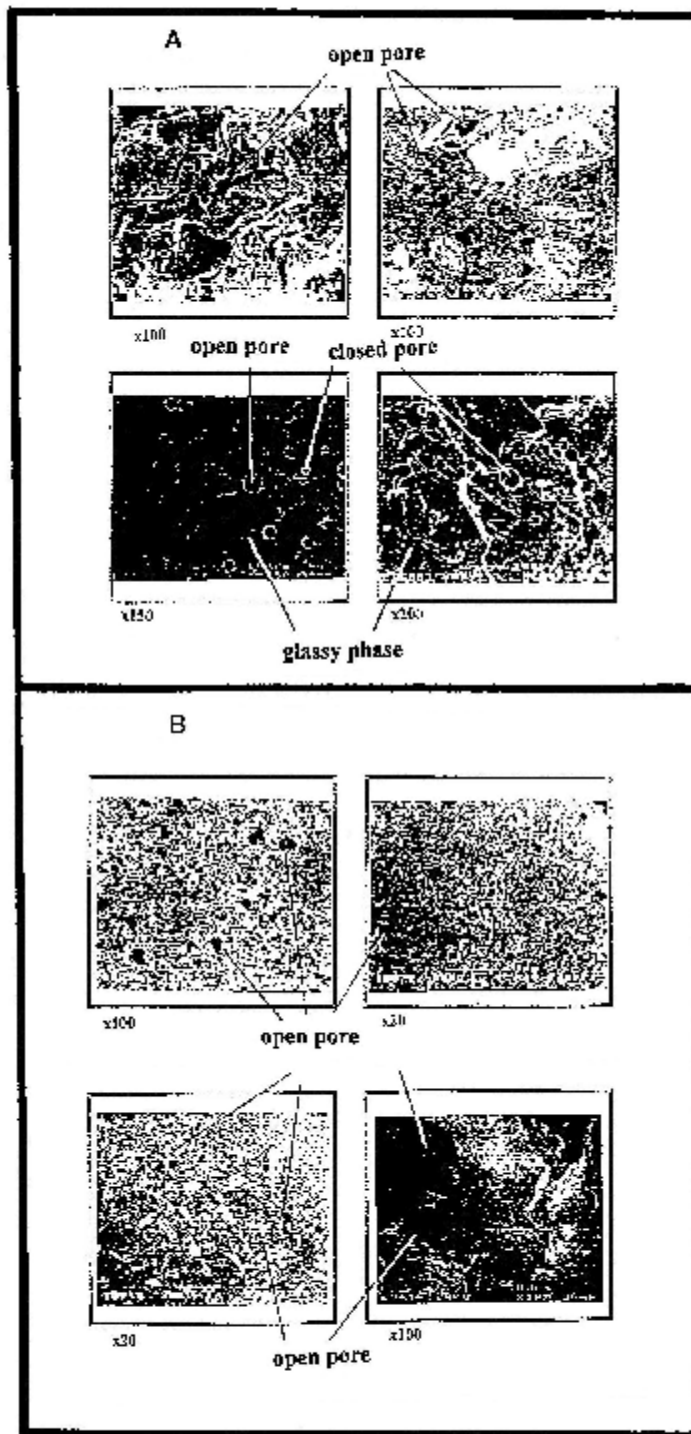


Fig. (10) SEM micrograph of ceramics prepared by (A) semi-dry pressing, (B) slip-casting.