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Preparation of fluorescent glasses with variable compositions

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Abstract

Results on exploration of making a large variety of different glasses, which are easy to make, have compositions distinguishable from each other, and are easy to be located in a complicated background, is reported. Porous glasses made by leaching a sodium borosilicate glass were used as the starting glass. The porous glasses were soaked in a solution containing soluble salts (mostly nitrates) of the necessary cations. When the pores in the porous glasses were filled with the solution, excess solution was removed and the glasses were heated at 650 °C for 3 h to decompose the salts in the pores into oxides. A group of salts are found useful for this purpose.

By impregnating a fluorescent agent into the porous glasses, the glasses are made fluorescent under ultra-violet light and easy to be located. UO_3, Eu_2O_3, and some laser dyes are found to be appropriate fluorescent agents for this purpose. The effects of the concentrations of some fluorescent agents in the impregnating solution on the fluorescence intensity of the impregnated glasses were studied. The oxides impregnated into the glasses are also found to have strong influence on the fluorescence intensity of the glasses impregnated with a fluorescent agent.

Keywords: B. Porosity; C. Optical properties; D. Glasses

1. Introduction

Glass bulks or spheres that are easy to make, distinguishable to each other by their composition, and are easy to be located in complicated environment are of special applications. The purpose of this work is to find a way to easily make a large variety of different glasses with compositions easy to control, and find a way to make the glasses easy to be located. A possible way to achieve this purpose is to impregnate the desired ions into porous glasses.

Impregnation of substances into porous glasses has been widely studied for special applications, such as gradient index glasses to be used as lenses or waveguiding patterns [1–3], catalysts [4], slow drug delivery [5], making nanocomposites [6], and so forth. The ions that have been successfully impregnated into a porous glass include Ba [2], Ru [6], Os [6], Er [7], Zr [8], Ti [3,9], C [10], Cs [11], K [11], Cd [12], Sn [13], Fe [13,14], Ni [14], Al [14], Se [15], Te [15], and others. These works indicates that it is possible to impregnate multi-components into a porous glass.

By soaking porous glasses in a solution containing salts of the desired ions, the pores in the porous glass will be filled with the solution. After the glass is dried and heated at a high temperature, the salts in the pores decompose to form oxides, which become the components of the glass. Control of the composition of the impregnated porous glass should be easy because the concentration of the solution can be easily controlled. This paper describes our work on exploration of the possibility to make such glasses.

2. Experiments

The raw materials we used in this experiment are listed in Table 1.

Porous bulk glass and glass spheres (90–212 μm in diameter) were made from Vycor[1] glass. The glasses were heat-treated at 565 °C for 3 h for phase-separation, and then leached in 1 M nitric acid to remove the boron-rich phase. The porous glasses prepared in this way are used as the base glass to make impregnated glasses.

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[1] A trade name of Corning Inc.

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Table 1
Raw materials used in this work

Raw materials	Purity (%)	Manufacturer
Base glass, Corning 7930	N/A[a]	Corning
$Ba(C_2H_3O_2)_2$	99	Avocado Research Chemical Ltd.
$Ca(NO_3)_2{\cdot}4H_2O$	99–103	Alfa
$Cd(NO_3)_2{\cdot}4H_2O$	98.5	Alfa
$Ce(NO_3)_3{\cdot}6H_2O$	99.5	Alfa
$Co(NO_3)_2{\cdot}6H_2O$	98.5	Alfa
CrO_3	99.9	Fisher
$Cu(NO_3)_2{\cdot}3H_2O$	98–102	Alfa
$Dy(NO_3)_3{\cdot}6H_2O$	99.9	Alfa
$Fe(NO_3)_3{\cdot}9H_2O$	98–101	Alfa
$Gd(NO_3)_3{\cdot}6H_2O$	99	Alfa
$La(NO_3)_3{\cdot}6H_2O$	99.9	Alfa
$Mg(NO_3)_2{\cdot}6H_2O$	98–102	Alfa
$Mn(NO_3)_2{\cdot}xH_2O$	99.98	Alfa
$Nd(NO_3)_3{\cdot}6H_2O$	99.9	Alfa
$Ni(NO_3)_2{\cdot}6H_2O$	>19.8% Ni	Alfa
$Pr(NO_3)_3{\cdot}6H_2O$	99.9	Alfa
$Sm(NO_3)_3{\cdot}6H_2O$	99.9	Aldrich
$Sr(NO_3)_2$	98	Alfa
$Y(NO_3)_3{\cdot}6H_2O$	99.9	Alfa
$Yb(NO_3)_3{\cdot}6H_2O$	99.9	Alfa
$Zn(NO_3)_2{\cdot}xH_2O$	99	Alfa
$ZrOCl_2{\cdot}8H_2O$	98	Fisher
Fluorescent agents		
$Eu(NO_3)_3{\cdot}6H_2O$	99.9	Alfa
$UO_2(NO_3)_2{\cdot}6H_2O$	99.9	Fisher
Blue marking ink, FN-2 V	N/A	Spectronics Corp.
Laser dye, R610	N/A	Exciton, Inc.
Laser dye, Kiton Red	N/A	Exciton, Inc.
Laser dye, DCM	N/A	Exciton, Inc.
Laser dye, LDS 698	N/A	Exciton, Inc.
Laser dye, NB690	N/A	Exciton, Inc.

[a] N/A indicates "not applicable" or "no data available".

Solutions used to impregnate ions into the porous glasses were made by dissolving the corresponding salts into distilled water. Impregnation was done by soaking the porous glasses in a solution for 12 h at room temperature. The excess solution in between the glass spheres was removed by vacuum filtration and that on the surface of a bulk glass was removed by blowing with compressed air. The impregnated glasses were then heated at 650 °C for 3 h to decompose the salts into oxides.

The concentration of the impregnated oxides in a glass sample was analyzed by Energy Dispersion Spectra (EDS) and by X-ray fluorescence spectra (XRF).

To make the glass easy to be located in a complicated background, fluorescent agents were used. By impregnating fluorescent agents into porous glasses, the glasses will shine under ultraviolet light and can be easily located. The fluorescent agents chosen for this purpose are listed in Table 1. The procedure for impregnating the fluorescent agents into the glasses was the same as that for impregnating the ions described above. For impregnation of UO_3 and Eu_2O_3, the glasses were heated in the same way as for impregnation of the other ions to decompose the salts into oxides. The glasses were not heated when impregnating the other fluorescent agents.

The fluorescence intensity of the glasses impregnated with the fluorescent agents was observed by using a black box with both long wavelength ultra-violet (UV) light (365 nm) and short wavelength ultra-violet light (254 nm). The relative fluorescence intensity was estimated visually by comparing the brightness of the glasses in the black box with both short and long wavelength UV lights on.

3. Results and discussion

3.1. Concentration levels of various oxides in glass

The idea of this work is to conveniently make glass beads or bulks containing oxides with different levels of concentration by impregnating the ions, which are in a solution, into porous glass beads or bulks.

To calculate the amount of each component impregnated into the porous glass beads or bulks, we assume that: (1) the pores in the glass beads or bulks are fully filled with solution during impregnation. (2) The solution remains in the pores after filtration and the compounds in the solution decompose to form oxides after firing. Based on these assumptions, the amount of an oxide impregnated into the glass can be estimated according to the concentration of the solution and the porosity of the porous glass beads or bulks.

$$A = \frac{\mathrm{PC}}{\mathrm{PC} + d(100 - P)} \times 100\%$$

where A is the weight percentage of the oxide in consideration in the impregnated glass, P the porosity (vol.%) of the leached porous glass, C the concentration of the solution (grams of oxide in one milliliter of solution), and d is the density of the leached porous glass.

The weight loss (weight percent) of the glass after leaching can be taken as an estimation of the porosity of the leached porous glass because the density of the components in the precursor glass is close to each other. Since the leached glass is almost pure SiO_2, the density of fused silica (2.2 g/cm^3) can be used as the density of the leached glass.

Although the pores in the glass can be nearly fully filled up with the impregnated oxides if the soaking-and-firing cycle is repeated on the same piece of glass for many times, it is more practical to get the wanted composition in a single cycle of soaking-and-firing. Since the density and the porosity of the

Table 2
Concentrations of some ions in single-component solutions and the estimated amount of the oxides in the impregnated glass for a single-cycle of impregnation

Ion	Concentration in single-component solution (gram of oxide per milliliter solution)	Amount of oxide in impregnated glass for a single-cycle impregnation (wt.%)
La	0.41	7.40
Nd	0.42	7.56
Gd	0.45	8.06
Mg	0.14	2.65
Fe	0.20	3.75

Table 3
Calculated and measured amount of different oxides in porous beads impregnated in a multi-components solution

Oxides	Calculated amount of oxides (wt.%)	Measured amount of oxides by EDAX (wt.%)			Average of measured amount	Measured value Calculated value
		Measurement 1	Measurement 2	Measurement 3		
MgO	0.45	0.89	0.88	0.95	0.91	2.00
Nd_2O_3	1.35	0.69	0.66	0.72	0.69	0.51
Fe_2O_3	1.28	1.21	1.11	1.19	1.17	0.91
Gd_2O_3	1.44	1.13	1.04	1.01	1.06	0.74
La_2O_3	1.31	1.41	1.21	1.31	1.31	1.00

Table 4
Calculated (Cal.) and analyzed (Ana.) amounts of oxides in impregnated glass beads (wt.%)

Oxide	Sample 1 (high concentration)		Sample 2 (middle concentration)		Sample 3 (low concentration)		Sample 4 (high concentration with higher Fe_2O_3)	
	Cal.	Ana.	Cal.	Ana.	Cal.	Ana.	Cal.	Ana.
MgO	0.54	[a]	0.11	[a]	0.011	[a]	0.45	[a]
Nd_2O_3	1.61	1.45	0.33	0.35	0.033	0.025	1.35	1.52
Fe_2O_3	0.77	1.12	0.16	0.47	0.016	0.21	1.28	1.85
Gd_2O_3	1.72	1.27	0.35	0.34	0.035	0.031	1.44	1.45
La_2O_3	1.57	1.34	0.32	0.37	0.032	0.035	1.31	1.37

[a] Not detected.

porous glass are constant in this experiment, the amount of an oxide that can be impregnated into the porous glass in a single cycle of impregnation is determined by the concentration of the solution. The highest concentration of each ion used in this experiment is near the saturated concentration of the ion. Lower concentrations for each ion are also used to get different concentration levels of the oxide in the impregnated glass. Table 2 gives the highest concentrations of some ions in single-component solutions and the estimated amount of the oxides in the impregnated glass for a single-cycle of impregnation.

When two or more ions are to be impregnated into a piece of porous glass, multi-component solutions are used. Multi-component solutions are made by simply mixing single-component solutions. Table 3 gives the calculated and measured amount of each oxide in porous glass beads soaked in a multi-component solution containing equal volume of single-component solutions of the ions with the highest concentration level. It can be seen that the measured amounts of the oxides diverge from the calculated amounts by a factor of two. The differences between the measured and calculated amounts may be caused by a couple of factors, including the errors in the concentration of the solutions and the porosity of the glass, the degree of filling to the pores by the solution, the amount of residual solution on the surface of the beads after filtration, etc. Although the measured amounts of the oxides are not very close to the calculated amounts, the data from the three different measurements are close to each other. This indicates that the beads have relatively uniform composition. If we use different concentrations of the solutions to impregnate the porous glass, we should be able to get glasses with compositions distinguishable from each other.

To make porous glasses with different composition by impregnation, concentration levels of oxides in impregnated glasses lower than those shown in Table 3 are needed. Since EDAX is not a good method for analyzing low concentrations, X-ray fluorescence analysis (XRF) is used in this research.

Table 4 gives the calculated and analyzed data of the amount of the oxides in different samples of impregnated glass beads. Since Mg was not detected by the XRF method, it cannot be used as a component to identify the impregnated glass. The three concentration levels of the other components can be clearly distinguished by the XRF method except for the middle and low concentration levels of Fe_2O_3. The measured concentration of Fe_2O_3 in the low concentration sample is obviously higher than the calculated one, possibly because the base glass contains some amount of Fe_2O_3. Thus, all the oxides except MgO can be used as components to identify an impregnated glass. There are four concentration levels for Gd_2O_3, La_2O_3, and Nd_2O_3 considering the 0 wt.% concentration level, while there are three concentration levels for Fe_2O_3.

According to the analyzed amounts of the oxides, the sample with the high concentration level and higher Fe_2O_3 can be distinguished from the one with the high concentration level. Therefore, by increasing the concentration of one of the oxide and keeping the concentrations of the others un-changed, it is possible to have a higher concentration level for one of the oxides in each glass sample.

Tables 5 and 6 give the calculated and analyzed amounts of some other oxides in some more samples. Since we consider the amount of each oxide in the base glass to be one of the concentration levels, we have four concentration levels for each of the oxides in Table 5 except CdO and Y_2O_3. The amounts of CdO and Y_2O_3 in the base glass are relatively high and are not

Table 5
Calculated (Cal.) and analyzed (Ana.) amounts of oxides in impregnated glass spheres (wt.%) (“as-received” indicates the glass spheres were not impregnated)

Oxide	Sample 5 (high concentration)		Sample 6 (middle concentration)		Sample 7 (low concentration)		As-received glass
	Cal.	Ana.	Cal.	Ana.	Cal.	Ana.	
CdO	1.51	0.64	0.26	0.32	0.043	0.120	0.046
CoO	0.79	0.32	0.13	0.11	0.022	0.022	0.001
CeO_2	1.38	0.72	0.23	0.21	0.039	0.058	0.013
MnO	0.93	0.48	0.16	0.14	0.026	0.029	0.002
NiO	0.81	0.32	0.14	0.12	0.023	0.026	0.002
Y_2O_3	0.97	0.28	0.16	0.13	0.027	0.055	0.030

Table 6
Calculated (Cal.) and analyzed (Ana.) amounts of oxides in impregnated glass spheres (wt.%) (“as-received” indicates the glass spheres were not impregnated)

Oxide	Sample 8 (high concentration)		Sample 9 (middle high concentration)		Sample 10 (middle low concentration)		Sample 11 (low concentration)		As-received glass
	Cal.	Ana.	Cal.	Ana.	Cal.	Ana.	Cal.	Ana.	
CaO	0.90	0.632	0.18	0.1775	0.036	0.014	0.007	0.060	0.05
Cr_2O_3	1.25	1.56	0.25	0.2905	0.050	0.0748	0.010	0.0407	0.0068
CuO	1.15	0.5981	0.23	0.0897	0.046	0.0171	0.009	0.0078	0.0009
SrO	1.15	0.2728	0.23	0.0509	0.046	0.0124	0.009	0.0082	0.0034
ZnO	1.07	0.3703	0.21	0.0547	0.043	0.0105	0.009	0.0056	0.0014
ZrO_2	0.97	0.9031	0.19	0.9746	0.039	0.8989	0.008	0.9429	0.8468

distinguishable from the amount in the low concentration sample (sample 7). Therefore, we only have three concentration levels for these two oxides.

The data in Table 6 show that ZrO_2 cannot be used as an component to identify impregnated glasses because the amount of ZrO_2 in the base glass is high. Since the CaO content in the base glass is higher than the calculated amount of in the middle low concentration sample (sample 10), we can only have three concentration levels in an impregnated porous glass.

The samples listed in Table 6 were prepared in an attempt to make five concentration levels of oxides in porous glasses. The data for Cr_2O_3, CuO, SrO, and ZnO indicates that it is possible to make five distinguishable concentration levels for these oxides.

From the above results, we can find that:

1. It is possible to make porous glasses with distinguishable compositions by impregnating a porous glass sample with a solution containing salts of the desired ions.
2. When the glass is impregnated with up to six ions (more ions are also possible) at a time, the oxides and their concentration are distinguishable by the XRF method.
3. Most of the oxides can have four or even five distinguishable concentration levels, and some other oxides can only have three concentration levels because the amounts of these oxides in the base glass are relatively high.
4. MgO and ZrO_2 cannot be used as components to identify an impregnated glass.

Table 7
Relative fluorescence intensity of glass beads impregnated with different fluorescent agents

UO_3 in glass (wt.%)	6	1.8	0.4	0.08	0.008
Relative fluorescence intensity[a]	50	80	95	95	95
Eu_2O_3 in glass (wt.%)	3	1	0.1	0.01	0.001
Relative fluorescence intensity[a]	60	30	20	20	10
FN-2V/acetone (v/v)	1/2	1/5	1/10	1/20	1/50
Relative fluorescence intensity[a]	100	100	98	95	90

[a] The fluorescence intensity of the porous glass beads impregnated in a solution of FN-2V/acetone = 1/5 was taken as 100.

3.2. Impregnation of fluorescent agents

The relative fluorescence intensity of porous glass beads impregnated with the fluorescent agents are given in Tables 7 and 8.

When UO_3 is used as fluorescent agent, lower concentration in this experimental range gives stronger fluorescence intensity. Since there is a so-called concentration quenching effect, higher concentration of UO_3 reduces the fluorescence intensity. For Eu_2O_3-impregnated beads, higher concentration gives stronger fluorescence intensity. Under the UV lights in this

Table 8
Relative fluorescence intensity and color of porous glass beads impregnated with laser dyes

Dye	10 mg in 10 ml methanol		10 mg in 100 ml methanol	
	Relative fluorescence intensity[a]	Color	Relative fluorescence intensity[a]	Color
R610	95	Orange	95	Bright orange
Kiton Red	90	Orange	90	Bright orange
DCM	40	Pale orange	40	Orange
LDS 698	40	Red	40	Orange-red
NB690	5	Dark red	5	Dark red

[a] The fluorescence intensity of the porous glass beads impregnated in a solution of FN-2V/acetone = 1/5 was taken as 100.

experiment, the relative fluorescence intensity of Eu_2O_3-impregnated beads is lower than that of the UO_3-impregnated beads.

The fluorescence intensity of the glass beads impregnated with FN-2V is very strong, but the color is not good for distinguishing the glass from the background because many organic fibers, like paper and clothes, in the environment shine the same color under UV light.

For the laser dyes used in this experiment, only R610 and Kiton Red have strong fluorescence under the UV lights described above. Very low concentration of the impregnating solution of these two dyes will make the glass beads brightly fluorescent. The other dyes may have strong fluorescence if excited with lights of other wavelength, but their fluorescence is weak under the UV lights in this experiment.

3.3. Influence of oxides on the fluorescence intensity of different fluorescent agents

To find the influence of each oxide on the fluorescence intensity of the different fluorescent agents, small amount of porous beads were impregnated in a solution containing a single component. After removing the excess solution and firing the impregnated beads at 650 °C for 3 h, a solution containing one of the fluorescent agents was used to impregnate the fired beads. The fluorescence intensity was then compared with each other. Table 9 gives the relative fluorescence intensity of the glass beads.

It is shown that all the oxides with strong color, such as CoO, Cr_2O_3, CuO, Fe_2O_3, MnO, and NiO, have strong effects of reducing the fluorescence intensity, no matter what fluorescent agent was used. Some oxides that have a light color, such as CeO_2, Nd_2O_3, and Pr_2O_3, reduce the fluorescence intensity to a different degree. None of the colorless or white oxides reduces the fluorescence intensity of glass beads impregnated with FN-2V and R610. The influences of the oxides on the fluorescence intensity of UO_3-impregnated glass beads are complicated. Except the influence of the color of the oxides, some colorless or white oxides also strongly reduce the fluorescence intensity of the UO_3-impregnated beads. This may be caused by the reaction between the oxides and the UO_3.

Table 9
Influence of different oxides on the fluorescence intensity of porous glass beads impregnated with uranium, FN-2V, and R610

Oxides impregnated in glass beads	Relative fluorescence intensity[a] of glass beads impregnated with different fluorescent agent		
	FN-2V	Uranium	R610
As-received beads	100	95	95
BaO	100	30	95
CaO	100	95	95
CdO	100	80	95
CeO_2	80	20	40
CoO	20	5	N/M[b]
Cr_2O_3	5	0	N/M
CuO	50	5	N/M
Dy_2O_3	100	30	95
Fe_2O_3	20	0	N/M
Gd_2O_3	100	80	95
La_2O_3	100	80	95
MnO	20	5	N/M
Nd_2O_3	100	50	70
NiO	30	10	N/M
Pr_2O_3	100	10	90
Sm_2O_3	100	20	95
SrO	100	80	95
Y_2O_3	100	90	95
Yb_2O_3	100	40	95
ZnO	100	95	95

[a] The fluorescence intensity of the porous glass beads impregnated in a solution of FN-2V/acetone = 1/5 was taken as 100.
[b] N/M indicated data were not measured.

4. Conclusions

By impregnating a porous glass sample with a solution containing salts of the desired ions followed by decomposing the salts into oxides at a high temperature, porous glasses with compositions distinguishable from each other can be made.

Six or more components can be impregnated into a single glass sample and the components and their amount can be analyzed by XRF with reasonable accuracy. These components and their amounts in the glass can be used to distinguish the glass from others.

Some fluorescent agents, such as UO_3, FN-2V, and R610, can be impregnated into porous glass to make the glass strongly fluorescent. Oxides with strong color greatly reduce the fluorescence intensity. These oxides cannot be used in fluorescent glasses as components for identifying the glass.

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