

Influence of carbon steel and its corrosion products
on the leaching of elements from a simulated waste
glass
(Research Document)

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Influence of carbon steel and its corrosion products on the leaching of elements from a simulated waste glass (Research Document)

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Abstract

The influence of carbon steel and its corrosion products on leaching of elements from simulated high level radioactive waste glass (P0798) has been investigated in batch-type experiments of up to one year at 25 ± 3 °C under argon atmosphere (<1 ppm oxygen) with a surface area of the glass sample to leachant volume ratios of 10, 100 and 1000m⁻¹. For each series, a polished glass cube or glass particles were immersed in double distilled deoxidized water with powder of carbon steel or of magnetite, as an assumed carbon steel corrosion product, in the ratio of 10 gram carbon steel (or magnetite) to 1 liter of water.

Normalized mass losses of Si, B, Na and Cs in the systems of glass-carbon steel-water and of glass-magnetite-water apparently increased compared with those in the glass-water system. The glass dissolution could be accelerated by increasing hydroxyl ion resulting from hydrolysis of carbon steel or of magnetite. The pH values of leachate become eventually almost constant and it indicates that a steady state has been attained between these hydroxyl ion releases and its consumption, which is a disruption process of siloxane bond by hydroxyl ion in solution. The influence of carbon steel and its corrosion products on leaching of elements from the waste glass can be predominantly attributed to a pH effect in this experiment. The increase of glass corrosion rate, however, could be assessed within one order as an influence of the existence of carbon steel and/or its corrosion products.

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模擬廃棄物ガラスからの元素浸出における炭素鋼およびその腐食生成物の影響
(研究報告)

佐竹憲治¹⁾、亀井玄人²⁾

要 旨

模擬廃棄物ガラス (P0798) からの元素の浸出に対する炭素鋼とその腐食生成物の影響が、窒素雰囲気 (<1 ppm 酸素) のもとでバッチ式試験でおこなわれた。期間は 1 年までで、温度を 25 ± 3 、浸出液体積に対するガラスの表面積の比を 10、100、および 1000m^{-1} に設定した。おのおのの試験において、研磨した立方体、あるいは粒子状のガラス試料を 2 段蒸留脱イオン水中に浸した。そして、これらに対して、炭素鋼または磁鉄鉱 (腐食生成物として仮定した材料) 粉末を、固体 : 液体 = 10g : 1ℓ となるよう、加えた。

ガラス - 炭素鋼 - 水系やガラス - 磁鉄鉱 - 水系での Si、B、Na、および Cs の規格化浸出量は、ガラス - 水系のそれらにくらべて明らかに増大した。このガラスの溶解は、炭素鋼や磁鉄鉱の加水分解による水酸化物イオンの増大ともなって加速されたものと考えた。浸出液の pH の値はいずれも最終的にはほぼ一定となったが、このことはこれら水酸化物イオンの放出と同時に、このイオンによるガラスのシロキサン結合の破壊がおり、放出と消費によってイオン濃度が定常状態になったものと考えた。

炭素鋼やその腐食生成物が廃棄物ガラスからの元素の浸出に及ぼす影響は主として pH 効果に帰着できると考えられる。しかしながら炭素鋼あるいはその腐食生成物の存在によるガラスの腐食速度への加速効果は 1 桁以内であると見積もられる。

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1. Introduction

The objective of this study is to clarify how carbon steel, a candidate material of container for the geological disposal of high level radioactive waste, and magnetite as an assumed corrosion product of the carbon steel accelerate the leaching of elements from a nuclear waste glass. With respect to the influence of metal or its corrosion products on glass corrosion, some experimental studies were previously reported: Bart et al. (1987) investigated the influence of steel corrosion products in experiments up to one year at 90 °C with a sample surface area to leachant volume ratio (SA/V) of 10 m⁻¹. They summarized that glass corrosion is enhanced by steel corrosion products and the quantity of corroded glass may be dependent on the SA and not on the total amount of steel corrosion products in the system, suggesting a mechanism controlled by silica sorption on the steel corrosion products. Lanza and Ronsecco(1982) conducted corrosion tests of borosilicate glass containing 20% in weight of simulated waste oxides in glass ampoules in which 30 mL of paste composing of 70% montmorillonite and 10% Fe₂O₃ at 50 and 80 °C up to a maximum of 60 weeks. They suggested that although the effect of Fe₂O₃ is not negligible, the use of Fe₂O₃ to simulate corrosion products is probably a crude approximation. McVay and Buckwalter(1982) tested the interactive effects between ductile iron and PNL 76-68 borosilicate glass in 90 °C static deionized, tuff, and basalt groundwaters, up to 28 days. They indicated that elemental removal rates in all of the leachates are initially similar to those observed for early leaching in deionized water without the presence of iron, but in the presence of iron, the leach rates do not decrease as rapidly with time as they do in deionized water alone.

All of these previous experiments were performed under atmospheric condition. As Lanza and Ronsecco(1982) mentioned, a more realistic approach is needed so that the authors carried out experiments under oxygen-free conditions using a glove box.

2. Procedures and experimental conditions

Static leaching tests were carried out in a laboratory. The experimental conditions are as follows:

(1) Specimens:

- Simulated waste glass (P0798, Kawamura et al., 1989)
- Carbon steel (JIS 3101 SS 41) powder
- Simulated corrosion products : Magnetite powder, Fe_3O_4 , purchased from Wako Chemicals Co.

Table 1 and 2 show the chemical composition of the simulated waste glass and of the carbon steel, respectively.

(2) Temperature:

Room temperature (25 ± 3 ° C)

(3) Atmosphere:

Argon gas (<1 ppm oxygen)

(4) Leachate:

Double distilled deoxidized water

(5) Material of reactor vessel

Teflon

(6) Duration of reaction

28, 91 and 364 days

(7) Glass surface area to solution volume ratio (SA/V):

SA/V=10 (A polished cubic glass, SA=6cm², was soaked in 60cm³ of water), 100 and 1000 m⁻¹ (Glass grains, the specific surface area = 134 cm²/g and the grain size=250~420· μ m, were immersed in 60cm³ of water).

For each series of experiment at the SA/V=10, 100 and 1000 m⁻¹, the following three systems were provided:

- Glass – water
- Glass - carbon steel powder – water (carbon steel/water ratio = 10g/L)
- Glass - magnetite powder - water (magnetite/water ratio = 10g/L)

3. Results and Discussion

Table 3-7 shows the data and Figure 1-4 show the variations of normalized elemental mass loss with lapsing time and with SA' t/V respectively.

3.1 Normalized elemental mass loss

(1) Silicon

There is no difference between the normalized mass loss of Silicon (NL_{Si}) from simulated waste glass in water and that from the glass in water with carbon steel until 90 days after the beginning of reaction. The NL_{Si} in glass-carbon steel-water system, however, is about 6 times in maximum compared with that of glass-water system at 364 days.

The NL_{Si} in solution containing magnetite is about 7 times to that of the glass-water system.

(2) Boron

There is little difference between the normalized mass loss of boron (NL_B) from simulated waste glass in water and that from the glass in water with carbon steel until 90 days after the beginning of reaction. The NL_B in glass-carbon steel-water system, however, is about 11 times in maximum compared with that of glass-water system at 364 days.

The NL_B in solution containing magnetite is about 11 times to that of the glass-water system.

(3) Sodium

There is little difference between the normalized mass loss of sodium (NL_{Na}) from simulated waste glass in water and that from the glass in water with carbon steel until 90 days after the beginning of reaction. The NL_{Na} in glass-carbon steel-water system, however, is about 7 times in maximum compared with that of glass-water system at 364 days.

The NL_{Na} in solution containing magnetite is about 10 times to that of the glass-water system.

(4) Cesium

There is little difference between the normalized mass loss of cesium (NL_{Cs}) from simulated waste glass in water and that from the glass in water with carbon steel until 90 days after the beginning of reaction. The NL_{Cs} in glass-carbon steel-water system, however, is about 14 times in maximum compared with that of glass-water system at

364 days.

The NL_{Cs} in solution containing magnetite is about 10 times to that of the glass-water system.

(5) Strontium

There is little difference between the normalized mass loss of strontium (NL_{Sr}) from simulated waste glass in water and that from the glass in water with carbon steel until 90 days after the beginning of reaction. The NL_{Sr} in glass-carbon steel-water system, however, is about 11 times in maximum compared with that of glass-water system at 364 days. The NL_{Sr} in solution containing magnetite is about 10 times to that of the glass-water system. There is little difference between NL_{Sr} from the glass in water and that in water containing magnetite.

Normalized mass loss of Si, B, Na and Cs in the systems of glass-carbon steel-water and glass-magnetite-water apparently increased in comparison with those in the glass-water system.

3.2 pH dependency on elemental leaching from the glass

Several studies have been reported in terms of influences of metal and/or metal corrosion products on waste glass corrosion: Mc Vay and Backwalter (1982) carried out static experiment at 90 °C, up to 28 days, on interaction between PNL76-68 borosilicate glass (PNL 76-68) and ductile iron under a condition of deionized water, groundwater obtained from tuffaceous aquifer and that from basaltic aquifer, respectively. The results suggests that total amount of leaching elements increase and this tendency is attributed to be the effect of removal of these elements from the solution with production of iron silicate precipitates. Bart et al. (1987) indicated that the glass corrosion rate increased with increasing the surface area of iron corrosion products after an one-year static experiment at 90 °C and at $SA(glass)/V = 10m^{-1}$. It has been concluded that the reason why the glass corrosion rate increase could be an adsorption of leached silica onto the surface of steel corrosion products. In this study, the aqueous silica concentrations were less than the detection limit (Table 5) at initial stages of experiment in case of $SA/V = 10$ and 100 with carbon steel reactions. This result could suggest that effects of iron silicate precipitate and/or silica adsorption on the surface of carbon steel as mentioned above.

On the other hand, in the latter stage ($SA/V=100$ and 1000) of experiment with carbon steel and in all experiment with magnetite the aqueous silica concentration

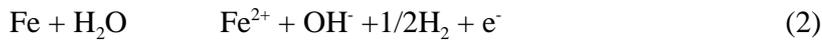
increased to be twice to seven times as much as that without carbon steel or magnetite (Table5, Fig.4). This result indicates that the predominant mechanism of the acceleration of glass corrosion rate could not be the iron silicate precipitates and/or silica adsorption.

Figure 5 shows variation of pH as a function of SA t/V. Table 8 also shows the data of pH values in all experiments. In the simple glass-water system, the pH of the solution is 5~6 at the beginning of the reaction and increases to be around 9 at SA t/V<10000 day m⁻¹ stages. Penetration of a ‘proton’ from water into the glassy network, replacing an alkali ion into solution (Paul, 1977) could be considered as a possibility of this pH increase. The glass corrosion process can be described as follows:

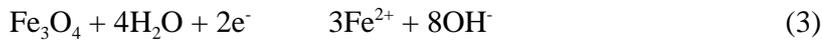


The pH values are higher in the solution with carbon steel or with magnetite than those without these materials, especially at the lower SA t/ V stages. This can be regarded as hydroxyl ion release assumedly expressed as follows in addition to the

reaction (1):



or



The pH values become almost constant after the stages at SA t/V>10000 day m⁻¹ for the all experiments. It is explained by the attainment of a steady state between these hydroxyl ion releases and their consumption that is a disruption process of siloxane bond by hydroxyl ion in solution:



Figure 6-8 shows the normalized mass loss of silicon (NL_{Si}) as a function of pH. The NL_{Si} increased about twice to five times with that the pH ascend from 9 to 10 except one series of experiment of which was conducted at SA/V=10m⁻¹ and in solution with carbon steel powder.

A simplified form of a general rate law for glass dissolution is expressed as

$$r = \text{SA} k a_{\text{H}^+}^- \quad (5).$$

where: r = rate at which glass is dissolved, SA=surface area, k=dissolution rate constant,

a_{H^+} =activity of H^+ and n =exponent of H^+ activity in solution. Knauss et al. (1990) gave -8.10 and 0.51 for the $\log k$ and n value, respectively, at $25^\circ C$ and at $pH > 7$ by flow-through experiments using a simple five-component borosilicate glass. Substituting these values for the equation (5), the rate rises 3.24 times with the pH increase from 9 to 10.

McGraile(1992) conducted a flow-through type experiment at $pH > 7$ using a simulated waste glass, P0798, and evaluated to be -4.69 ± 0.20 and -0.358 ± 0.02 for $\log k$ and n at $50^\circ C$, -1.93 ± 0.18 and -0.216 ± 0.02 for $\log k$ and n at $90^\circ C$, respectively.

There is a good agreement between this ratio and the measured increase of NL_{Si} , 2 ~ 5 times with increase from pH 9 to 10. Therefore, the influence of carbon steel and its corrosion products to leaching of elements from the waste glass can be predominantly attributed to a pH effect, which refer to hydroxyl ion release caused by a hydrolysis of steel or of its corrosion product. The increase of glass corrosion rate could be assessed within one order as an influence of the existence of steel and/or steel corrosion products.

4. Conclusion

Normalized mass losses of silicon, boron, sodium and cesium in solution with a presence of carbon steel or of magnetite increased compared with those in solution without these materials. The glass dissolution could be accelerated with increasing hydroxyl ion resulting from hydrolysis of carbon steel or magnetite. The pH values of leachate become eventually almost constant and it indicates that a steady state has been attained between these hydroxyl ion release and consumption, which is a disruption process of siloxane bond by hydroxyl ion in solution.

The influence of steel and steel corrosion products to leaching of elements from waste form glass is mainly attributed to a pH effect. The increase of glass corrosion rate, however, could be estimated within one order as an influence of the presence of steel and/or steel corrosion products.

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Table1 Chemical composition of PNC reference glass

Simulated waste glass	P0798
SiO ₂	46.60 %
B ₂ O ₃	14.20
Al ₂ O ₃	5.00
Li ₂ O	3.00
CaO	3.00
ZnO	3.00
Na ₂ O	10.00
P ₂ O ₃	0.30
Fe ₂ O ₃	2.04
Cr ₂ O ₃	0.10
NiO	0.23
Rb ₂ O	0.11
Cs ₂ O	0.75
SrO	0.30
BaO	0.49
ZrO ₂	1.46
MoO ₃	1.45
MnO ₂	0.37
RuO ₂	0.74
Rh ₂ O ₃	0.14
PdO	0.35
Ag ₂ O	0.02
CdO	0.02
SnO ₂	0.02
SeO ₂	0.02
TeO ₂	0.19
Y ₂ O ₃	0.18
La ₂ O ₃	0.42
CeO ₂	3.34
Pr ₆ O ₁₁	0.42
Nd ₂ O ₃	1.38
Sm ₂ O ₃	0.29
Eu ₂ O ₃	0.05
Gd ₂ O ₃	0.02
Total	100.00

**Table2 Chemical composition of carbon steel
and simulated corrosion products**

Carbon Steel Powder	
Fe	99.53
C	0.002
Si	0.02
Mn	0.21
P	0.017
S	0.015
Cu	0.01
Ni	0.01
Cr	0.03
O	0.15

Table3 Concentration(g/m³) of Cs in the leachant

SA/V=10m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powdeer(10g/l)	Glass only
28	2.00E-02	3.00E-02	Not detected
28	3.00E-02	4.00E-02	Not detected
28	2.00E-02	4.00E-02	Not detected
91	Not detected	6.00E-02	Not detected
91	2.00E-02	6.00E-02	Not detected
91	Not detected	6.00E-02	Not detected
364	2.90E-01	3.40E-01	4.00E-02
364	3.20E-01	3.40E-01	4.00E-02
364	4.50E-01	3.30E-01	5.00E-02

SA/V=100m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powdeer(10g/l)	Glass only
28	7.00E-02	1.80E-01	3.00E-02
28	8.00E-02	2.10E-01	3.00E-02
28	5.00E-02	1.70E-01	3.00E-02
91	5.30E-01	4.40E-01	No experiment
91	5.40E-01	4.60E-01	No experiment
91	4.10E-01	4.40E-01	No experiment
364	1.4	9.00E-01	8.00E-02
364	1.4	9.50E-01	1.30E-01
364	1.4	8.70E-01	1.60E-01

SA/V=1000m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powdeer(10g/l)	Glass only
28	2.30E-01	5.10E-01	1.30E-01
28	3.40E-01	7.20E-01	1.20E-01
28	2.10E-01	4.60E-01	1.40E-01
91	1.4	7.10E-01	No experiment
91	1.6	8.10E-01	No experiment
91	1.6	7.20E-01	No experiment
364	1.9	8.00E-01	5.00E-01
364	1.9	7.50E-01	9.30E-01
364	1.7	1.7	6.40E-01

Table4 Concentration(g/m³) of Na in the leachant

SA/V=10m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	1	3	0.9
28	0.8	3.1	9.5
28	0.9	3	1.3
91	0.4	3.4	0.4
91	0.5	3.4	0.6
91	0.4	3.3	0.5
364	2.8	5.9	0.6
364	2.9	5.9	0.6
364	4.4	5.6	0.8

SA/V=100m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	0.8	4	0.7
28	0.8	4.2	0.5
28	0.6	4	0.5
91	5.3	6.8	No experiment
91	5.2	6.9	No experiment
91	3.9	6.7	No experiment
364	16	13	1.3
364	17	15	1.5
364	17	13	1.7

SA/V=1000m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	2.6	10	1.6
28	3.4	10	1.6
28	2.4	8.9	1.8
91	15	18	No experiment
91	18	13	No experiment
91	18	14	No experiment
364	41	32	7.5
364	39	34	7.3
364	46	38	8.6

Table5 Concentration(g/m³) of Si in the leachant

SA/V=10m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	0.5	0.5	0.4
28	0.5	0.7	0.4
28	Not detected	1	0.7
91	Not detected	1	0.3
91	Not detected	1	0.6
91	Not detected	0.9	Not detected
364	Not detected	4.6	0.9
364	Not detected	4.4	1
364	1	4.4	1.2

SA/V=100m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	Not detected	2.5	Not detected
28	Not detected	3.1	0.7
28	Not detected	2.7	0.6
91	0.7	5.3	No experiment
91	0.9	6.4	No experiment
91	0.5	7.3	No experiment
364	9.5	12	1.9
364	7.9	14	2.9
364	11	15	3.7

SA/V=1000m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	2	11	2.2
28	2.9	11	2.3
28	2.1	9.5	3
91	8.2	17	No experiment
91	10	11	No experiment
91	9.6	14	No experiment
364	20	26	9.6
364	21	27	9.2
364	23	26	10

Table6 Concentration(g/m³) of B in the leachant

SA/V=10m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	Not detected	Not detected	Not detected
28	Not detected	0.2	Not detected
28	Not detected	0.2	Not detected
91	Not detected	0.5	Not detected
91	Not detected	0.5	0.2
91	Not detected	0.4	Not detected
364	1.5	2.1	0.2
364	1.6	2	0.2
364	2.7	2	0.3

SA/V=100m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	0.3	1.1	Not detected
28	0.3	1.3	Not detected
28	0.2	1.1	Not detected
91	3	2.7	No experiment
91	3.1	2.8	No experiment
91	2.1	3	No experiment
364	10	6.4	0.5
364	11	7.4	0.8
364	11	7.4	0.9

SA/V=1000m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	1.3	5.1	0.6
28	1.9	5	0.6
28	1.2	4.3	0.8
91	9.4	10	No experiment
91	11	6.5	No experiment
91	11	7.7	No experiment
364	26	21	4.4
364	26	22	4.3
364	31	24	5.2

Table7 Concentration(g/m³) of Sr in the leachant

SA/V=10m³

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	7.00E-03	Not detected	Not detected
28	1.10E-02	Not detected	3.50E-02
28	7.00E-03	Not detected	Not detected
91	1.50E-02	Not detected	8.00E-03
91	1.90E-02	7.00E-03	1.60E-02
91	8.00E-03	9.00E-03	1.60E-02
364	6.50E-02	Not detected	8.00E-03
364	7.10E-02	Not detected	9.00E-03
364	1.10E-01	Not detected	1.10E-02

SA/V=100m³

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	1.40E-02	Not detected	9.00E-03
28	1.50E-02	Not detected	8.00E-03
28	1.20E-02	Not detected	6.00E-03
91	1.70E-01	Not detected	No experiment
91	1.70E-01	Not detected	No experiment
91	1.30E-01	Not detected	No experiment
364	2.40E-01	3.50E-02	1.70E-02
364	2.50E-01	3.30E-02	3.50E-02
364	2.50E-01	1.60E-02	4.40E-02

SA/V=1000m³

Day	G + C.S. powder(10g/l)	G + Mt powder(10g/l)	Glass only
28	4.80E-02	6.00E-03	1.90E-02
28	6.40E-02	7.00E-03	2.10E-02
28	4.90E-02	6.00E-03	2.60E-02
91	2.20E-01	2.10E-02	No experiment
91	2.20E-01	1.30E-02	No experiment
91	2.20E-01	9.00E-03	No experiment
364	1.40E-01	3.30E-02	3.20E-02
364	1.40E-01	3.30E-02	3.60E-02
364	9.50E-02	1.00E-01	4.50E-02

Table8 pH Values of leachateSA/V=10m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powdeer(10g/l)	Glass Only
28	7.74	8.80	6.33
28	8.84	8.92	6.81
28	6.86	8.80	5.28
91	10.54	10.47	6.33
91	10.66	10.52	8.64
91	10.68	10.55	6.86
364	9.96	9.65	8.95
364	9.89	9.72	8.67
364	10.04	9.66	9.04

SA/V=100m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powdeer(10g/l)	Glass Only
28	9.31	9.56	7.59
28	9.47	9.60	9.03
28	9.48	9.59	8.97
91	10.69	9.66	No experiment
91	10.65	9.66	No experiment
91	10.51	9.55	No experiment
364	10.05	9.42	8.76
364	10.10	9.50	8.87
364	10.08	9.64	9.28

SA/V=1000m⁻¹

Day	G + C.S. powder(10g/l)	G + Mt powdeer(10g/l)	Glass Only
28	9.47	9.53	9.18
28	9.82	9.48	9.27
28	9.84	9.53	9.29
91	10.50	9.66	No experiment
91	10.53	9.84	No experiment
91	10.54	9.63	No experiment
364	10.69	9.78	9.26
364	10.65	9.80	9.80
364	10.51	9.96	9.82

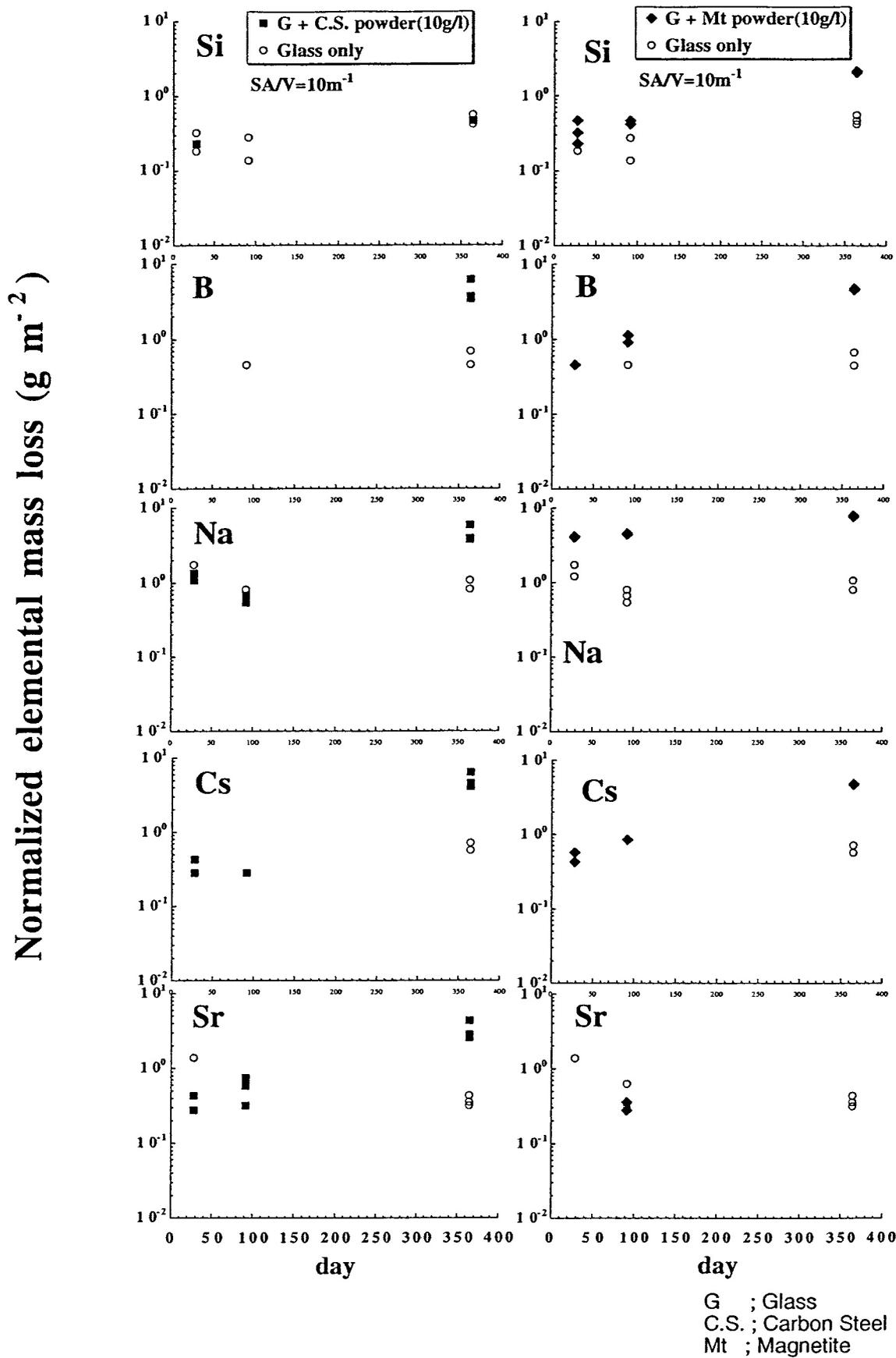


Figure1 Variation of normalized elemental mass loss with lapsing time at 10m^{-1}

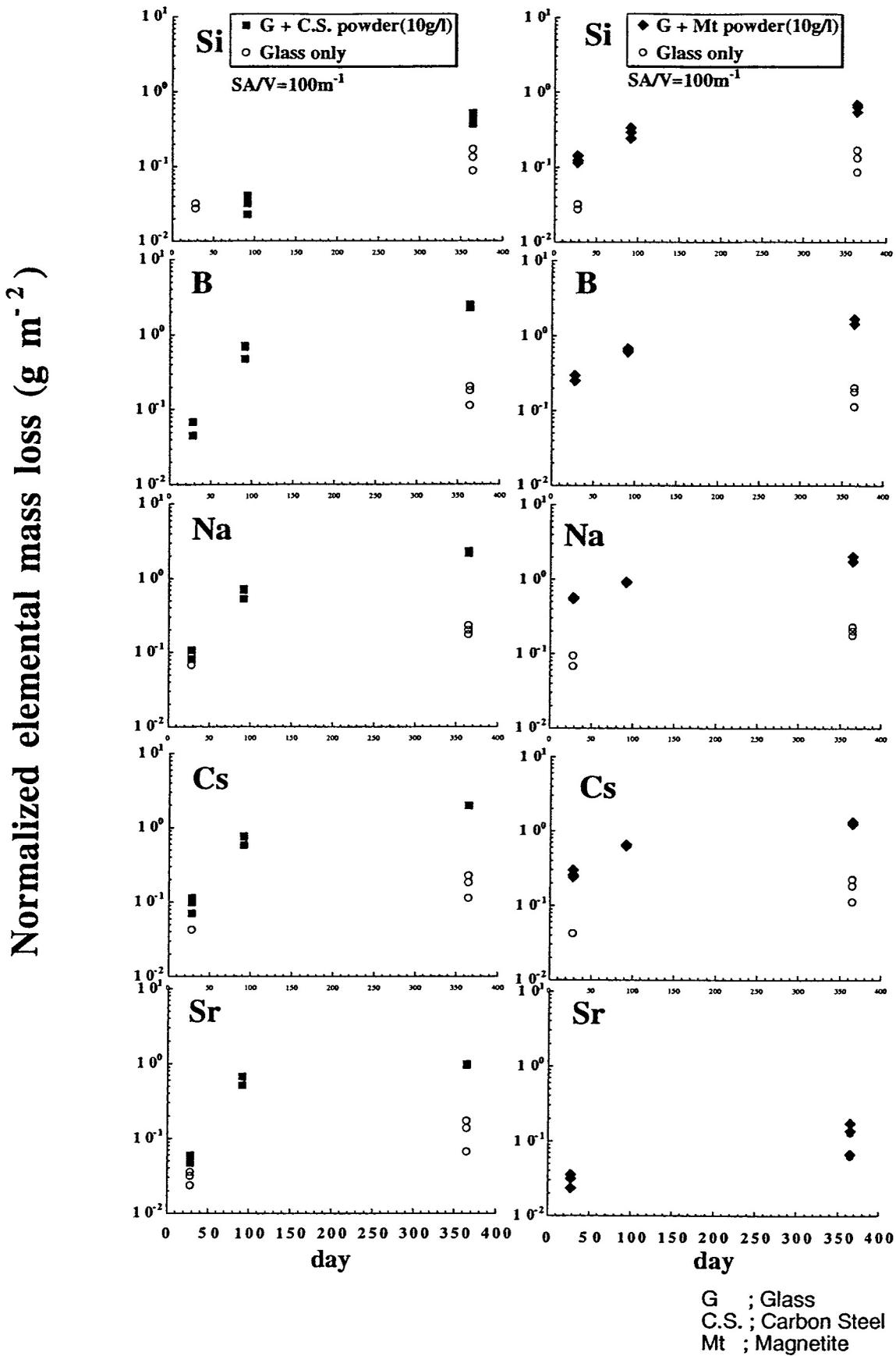


Figure2 Variation of normalized elemental mass loss with lapsing time at 100m^{-1}

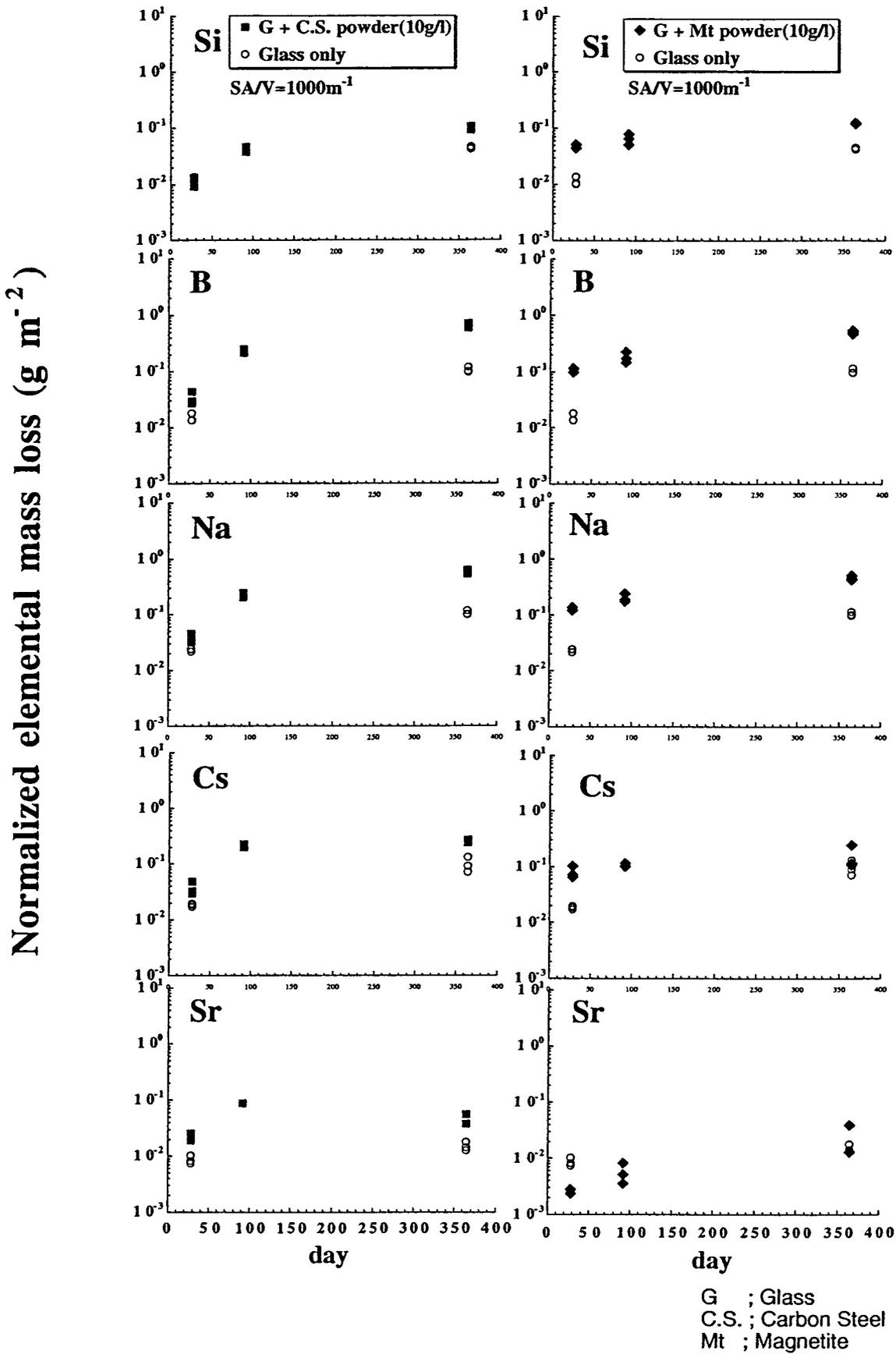


Figure3 Variation of normalized elemental mass loss with lapsing time at 1000m^{-1}

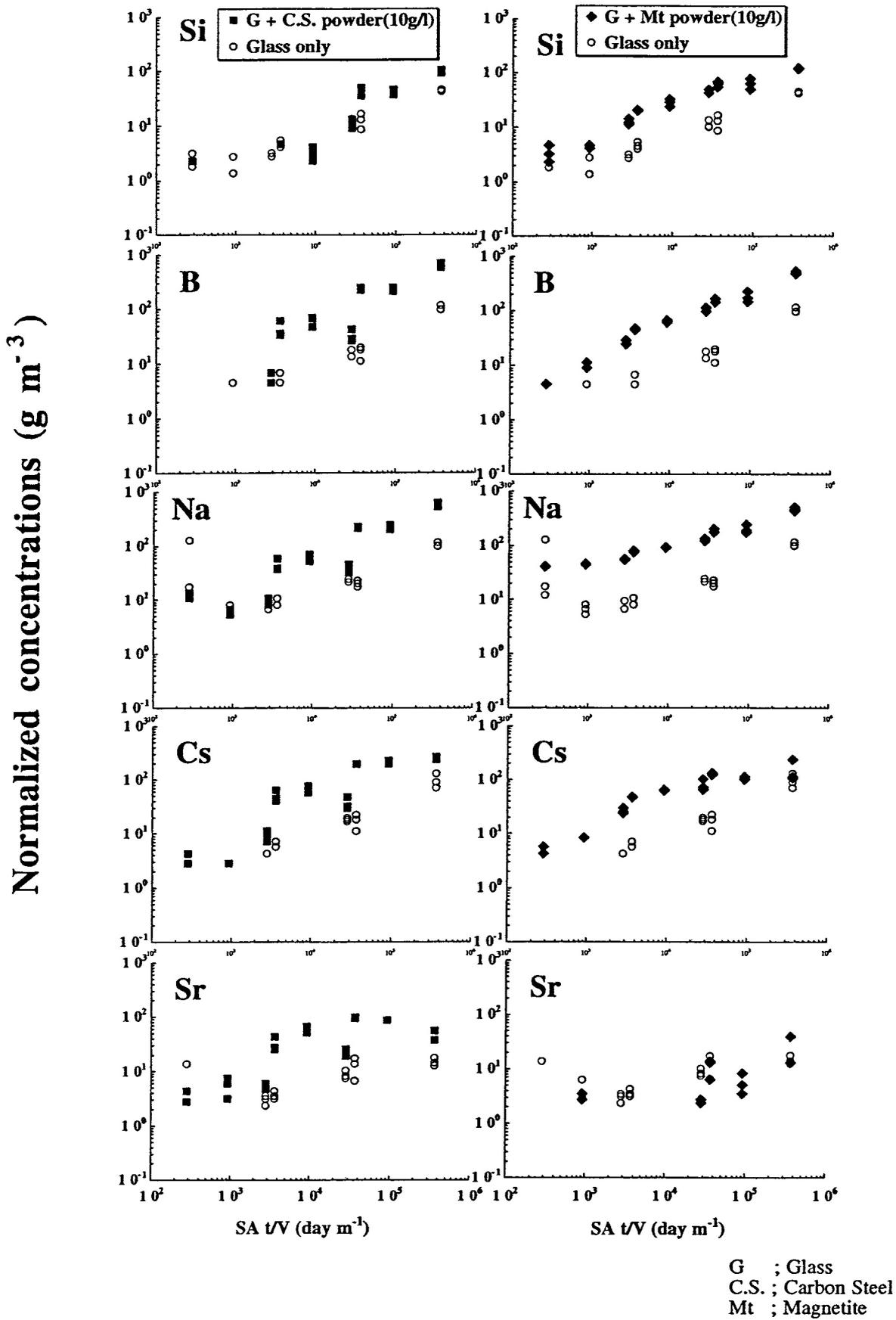


Figure4 Variations of normalized elemental mass loss as a function of SA t/V

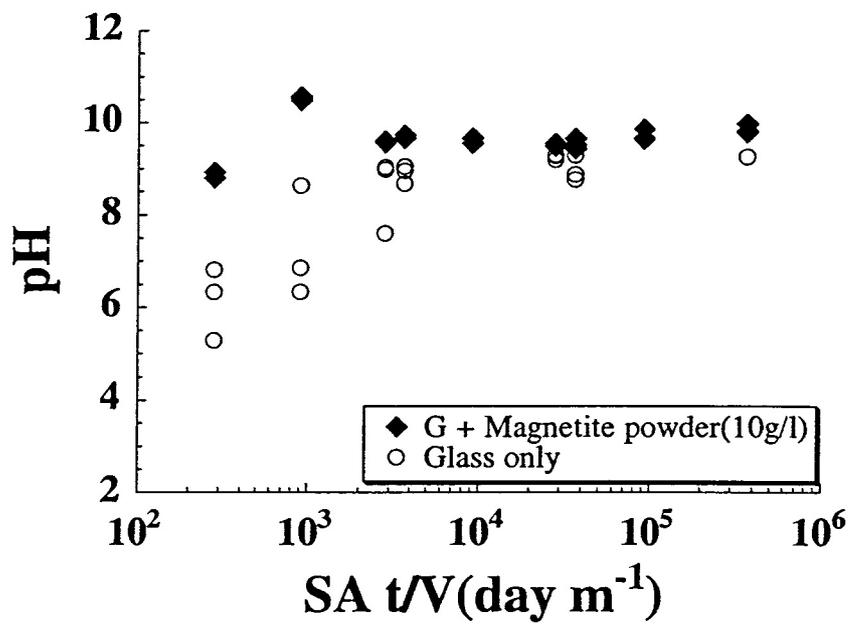
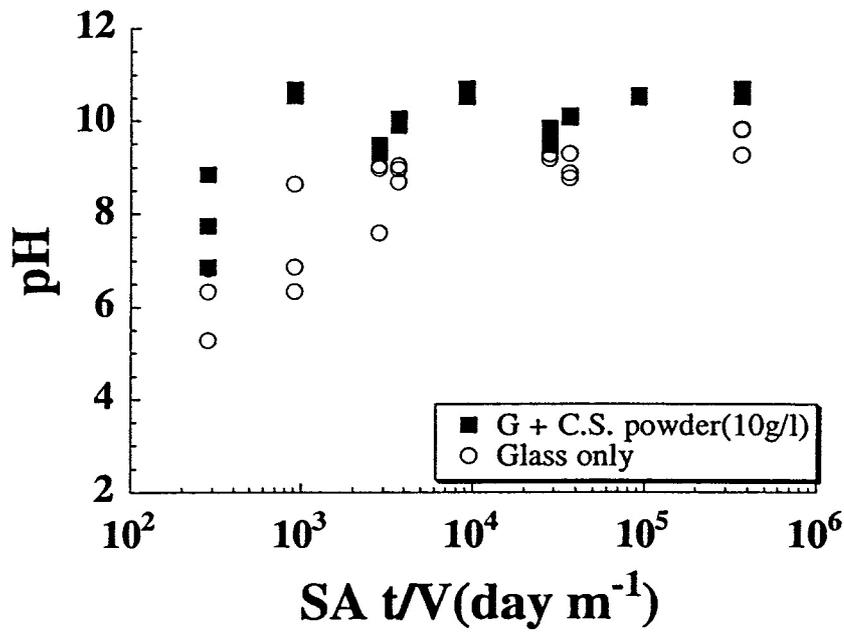


Figure5 Variations of pH as a function of SA t/V.

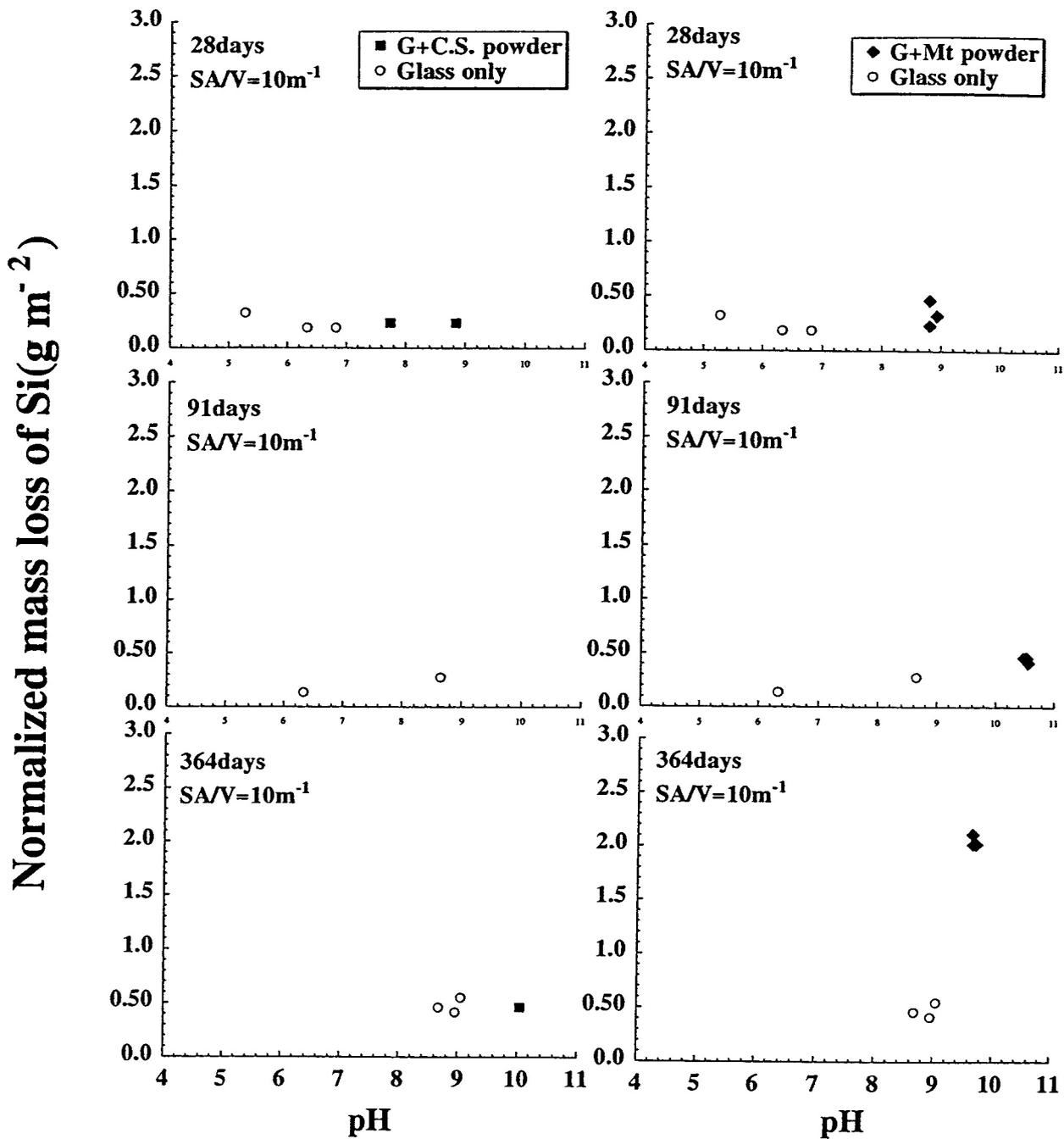


Figure6 Variations of normalized mass loss of silicon as a function of pH at SA/V=10m⁻¹

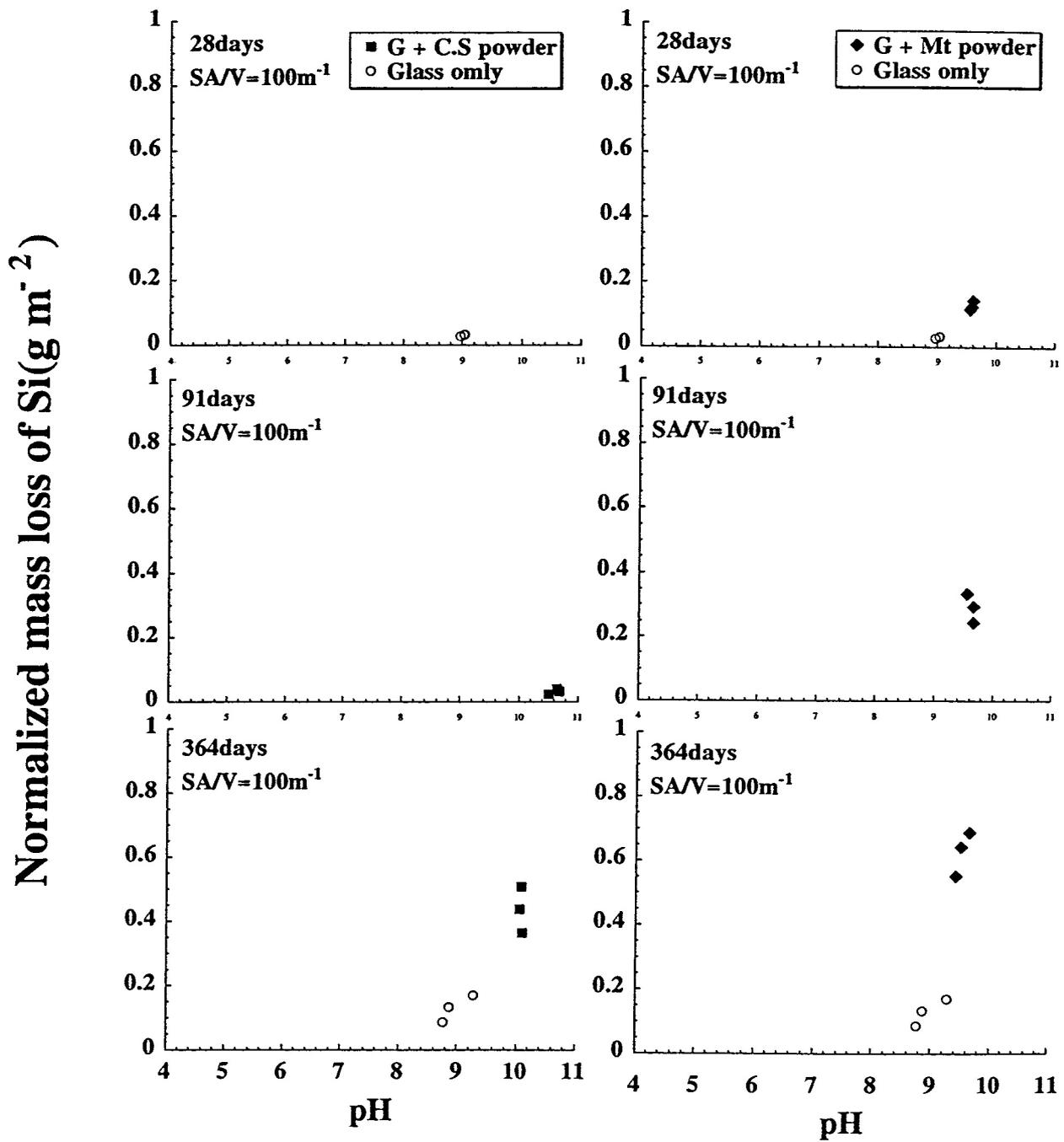


Figure7 Variations of normalized mass loss of silicon as a function of pH at SA/V=100m⁻¹

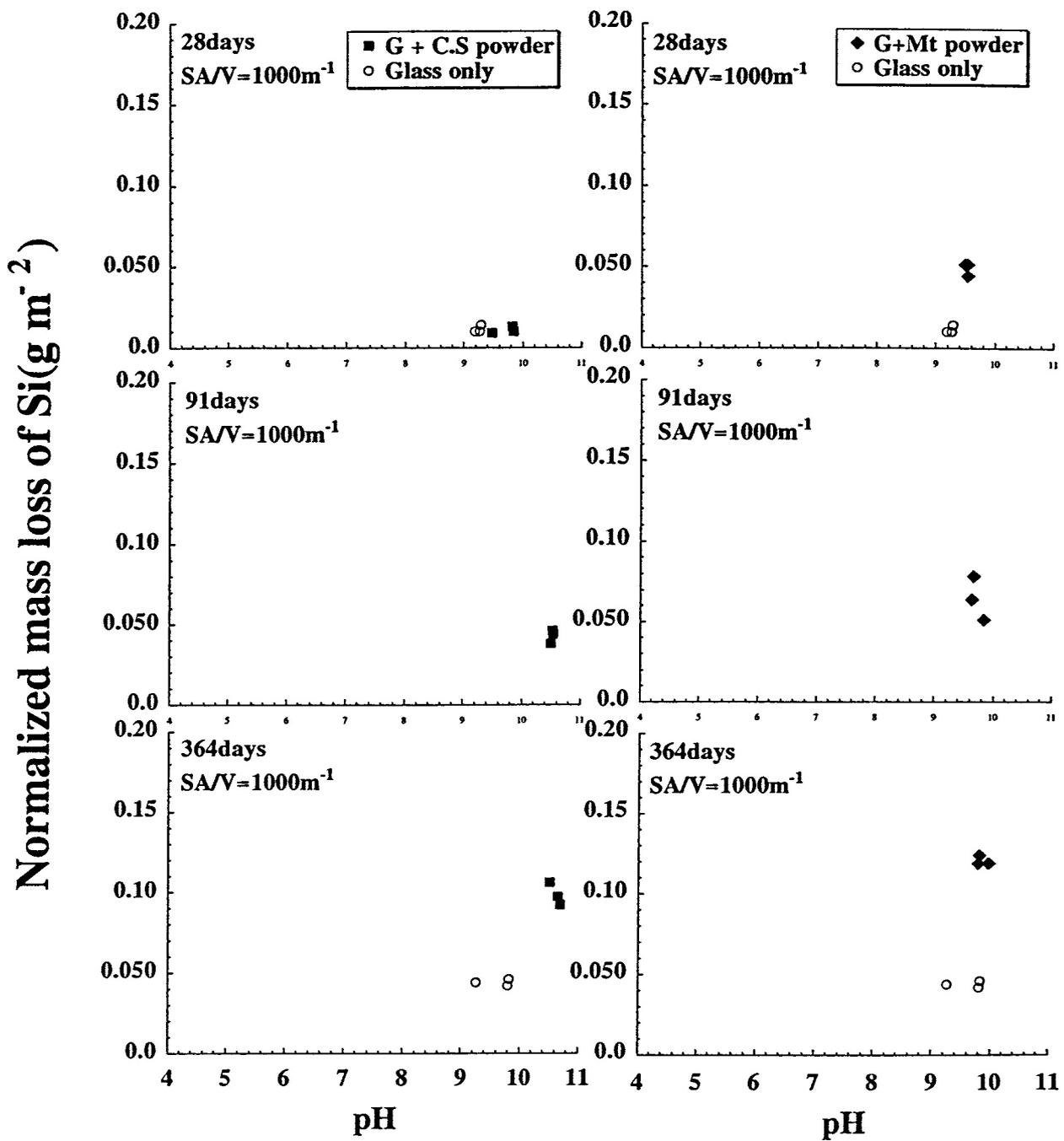


Figure 8 Variations of normalized mass loss of silicon as a function of pH at $\text{SA/V} = 1000 \text{ m}^{-1}$