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Parameters Affecting the Color Mechanism of Manganese Containing Colored Glasses

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Abstract: Color is an important tool for glass industry to let the product gain additional aesthetic and functional properties forced by the market demands. In this context, it is necessary to understand the color forming mechanism better in order to get target color and improve the yield. A detailed experimental study to investigate the relationship between the manganese content on color formation and the redox condition was carried out. All experiments were carried out at different level of iron and manganese oxides. Color parameters (dominant wavelength, brightness (%), purity (%) and L-a-b values) were examined for large interval of batch redoxes. In this paper, the results of above mentioned experimental studies were discussed together.

Key words: Colored glass, manganese oxide, iron oxide, batch redox.

1. Introduction

Manganese is one of the oldest and best known compounds which are used as colorants in glass. Besides, it is also used as a decolorant since it oxides iron and compensates the color formed by it. Manganese is usually added to the glass batch as pyrolusite, in the form of MnO_2. Having five common oxidation states, Mn^{7+}, Mn^{6+}, Mn^{4+}, Mn^{3+} and Mn^{2+}, polyvalent manganese occurs only in bivalent and trivalent forms in soda-lime-silica glasses. At higher temperatures, other oxidation states of manganese are unstable [1, 2].

It is also stated that Mn^{2+}, Mn^{3+} and Mn^{4+} cations or a mixture of those can be expected in glasses. Mn^{2+} and Mn^{3+} act as modifying cations, whereas, Mn^{4+} takes part in the network structure of silicate glasses with Si^{4+}. When manganese amount in the glass is increased by means of adding pyrolusite, the glass gets rich in oxygen, thus, manganese gets a higher oxidation degree. However, determination of only Mn^{2+} and Mn^{7+} is possible by analytical methods, Mn^{3+} and Mn^{4+} can be specified as an aggregate [3, 4].

In glass, Mn^{2+} and Mn^{3+} are usually present in an equilibrium depending mainly on the melting conditions. At high melting temperatures, and since Mn^{3+} is very sensitive to reducing conditions in the atmosphere and to reducing agents in the batch, the equilibrium of Mn^{2+} and Mn^{3+} ions is shifted strongly in favor of divalent form. Mn^{2+} containing glasses are obtained when melting is conducted in an oxidizing manner with the presence of reducing substances in the batch in order to prevent Mn^{2+} from becoming oxidized with oxygen from the furnace atmosphere. The linear absorption coefficient of Mn^{2+} is 100 times less than that of Mn^{3+} [5]. The absorption of MnO_2 containing glasses as a function of temperature and atmosphere is presented in Fig. 1 [6]. 1% MnO_2 containing glass has higher absorption in oxidizing conditions and its absorption increases with temperature. Oxidizing form has absorption in the visible range (380 nm to 780 nm), whereas reducing one does not. This information is very useful not only for coloring, but also in production process.

Mn^{2+} containing glasses are colorless, but change color from faintly yellow to brown while cooling. The absorption of divalent manganese occurs between 408 nm and 425 nm, whereas trivalent form has an absorption maximum from 490 nm to 500 nm and from 670 nm

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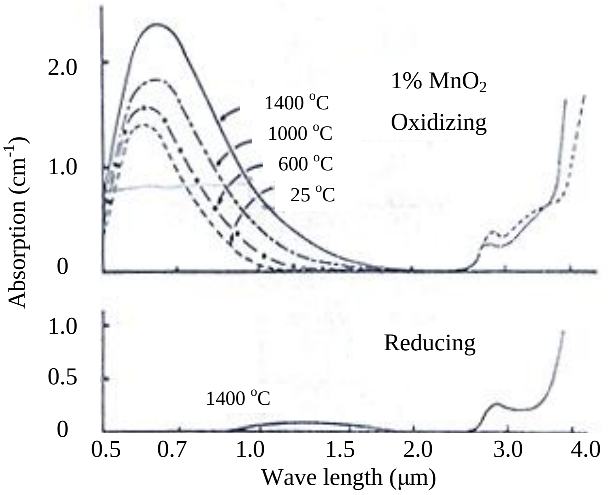


Fig. 1 Absorption of 1% MnO_2 containing glasses as a function of temperature and atmosphere.

to 710 nm. This means that in oxidizing conditions, glasses containing Mn^{3+} give an intense violet-amethyst color. Besides, Mn^{3+} increases the absorption of light in the ultraviolet region slightly, however, Mn^{2+} has no effect on the transmittance in the ultraviolet and infrared regions of the spectrum, but produces a green or orange fluorescence [2].

Considering that Mn^{2+} and Mn^{3+} are in equilibrium in the glass medium, Mn^{3+} can be determined theoretically by spectrometric methods, i.e., by Beer-Lambert law [7].

Beer-Lambert law states that:

$$\log (I/I_0) = -acd \qquad (1)$$

where, a is the absorption coefficient, c is concentration, d is the thickness in centimeters and I is the reduced intensity of the entering intensity, I_0 after passing through the specimen.

For manganese, this can be written as:

$$Mn^{3+} \text{ theoretical (spectrometric)} = \frac{-\log[\frac{T\,(@\,490\,\text{nm})}{92}]}{\text{sample thickness} \times \alpha\,(\text{Mn}\,@\,T\,490\,\text{nm})} \qquad (2)$$

where, T is transmission at 490 nm, α is absorption coefficient of Mn^{3+} at 490 nm, that is 4.03 [5], and sample thickness is in centimeters.

As mentioned before, manganese can be used as a colorant or decolorant in glass production. Depending on the amount in composition, it gives a pinkish color in soda-lime-silica matrix and with higher amounts purple and black colors are obtained. Besides, manganese is used to oxidize Fe^{2+} in order to get rid of the bluish green shade in flint glass under suitable conditions, therefore, manganese has been used as a decolorant almost from the beginning of glass production.

The linear optical absorption coefficients of Fe^{2+}, Fe^{3+} and Mn^{3+} in soda-lime-silica glasses, as optical density per centimeter per percentage by weight, are given in Fig. 2 [5]. Mn^{3+} has the highest absorption at 490 nm where it absorbs green light, thus pink/purple color is seen by the observer. Fe^{2+} and Fe^{3+} have absorption maximums at 1,050 nm and 380 nm, forming bluish green and yellowish green colors, respectively. With increasing amount of Fe_2O_3, the violet color of manganese gets lighter and a yellow-brown color is formed [1].

In order to evaluate the interaction of two polyvalent elements in a glass composition and to understand the effect of other ionic coloring elements on the oxidation state of manganese, the series of oxidation potential by Kocik [8] is used. The sequence of metal ions in order of increasing ease of oxidation is shown in Table 1 [5]. Table 1 indicates that oxides of elements situated above a given element will convert the one that is below it to a higher oxidation state and vice versa. Only chromium is capable of shifting the Mn^{2+}-Mn^{3+} equilibrium towards Mn^{3+} ion and all elements below manganese in the sequence decrease the violet color produced by Mn^{3+} ion [1].

Standard enthalpy per mole ($\Delta H^o/n$) values of the non-isothermal redox series of manganese and iron in soda-lime-silica glasses by Müller-Simon [9] are given below:

$$\frac{Mn^{2+}/Mn^{3+}}{37-71\ \text{kJ/mol}} \qquad \frac{Fe^{2+}/Fe^{3+}}{81-121\ \text{kJ/mol}}$$

Even in the existence of a small amount of MnO_2 in a glass containing Fe_2O_3, MnO_2 will convert Fe^{2+} to Fe^{3+}. This phenomenon is used for decolorization of iron containing flint glass.

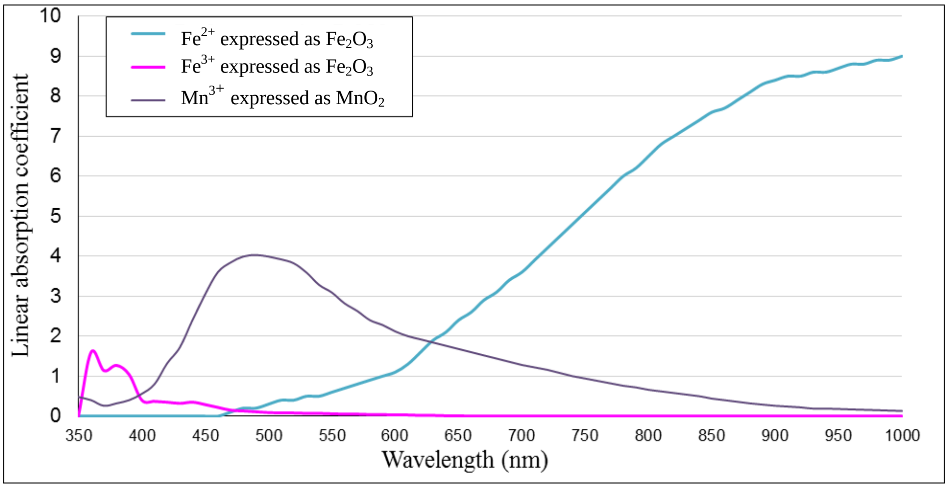


Fig. 2 **Linear absorption coefficients of Fe^{2+}, Fe^{3+} and Mn^{3+} in soda-lime-silica glasses (optical density per cm path length per percentage by weight oxide).**

Manganese reacts with iron according to Reaction (3), forming both Mn^{2+} and Mn^{3+} ions when the iron content of the glass batch is high.

$$Fe_2O_3 + 3MnO_2 + 1/2O_2 \rightarrow 2FeO + Mn_3O_4 + 2O_2 \tag{3}$$

In the presence of both manganese and iron ions, according to their sequence in Table 1 and $\Delta H^o/n$ values, Mn^{3+} is reduced to Mn^{2+} ion and the violet color is suppressed. When Fe_2O_3 content is lower than 0.1% by weight, manganese is used as a decolorant in glasses. Mn^{3+} ion acts as a decolorizer according to electron exchange Reaction (4) by oxidizing Fe^{2+} ion to Fe^{3+}.

$$Mn^{3+} + Fe^{2+} \rightarrow Mn^{2+} + Fe^{3+} \tag{4}$$

When exposed to direct sunlight, glasses decolorized by manganese get solarized and turn to brown in time.

Oxidation of Mn^{2+} ion in UV radiation occurs according to Reaction (5):

$$Mn^{2+} + Fe^{3+} \rightarrow Mn^{3+} + Fe^{2+} \tag{5}$$

Besides the disadvantage of solarization of the glass products, strict control of the oxidation level of glass batch is necessary since manganese and iron combination in glass is very sensitive to the furnace atmosphere [1]. In industry, the oxidation level of the glass is adjusted by adding redox materials such as carbon, sulphur, arsenic, sulphates and nitrates to the batch. The oxidation level of a glass batch is indicated by the batch redox number which is a semi-empirical scale [10].

The redox numbers for different materials which are listed in Table 2 are determined experimentally by Appleby Calumite Ltd. [11]. Here the redox number of MnO_2 is given as +1.09 which is higher than Fe_2O_3, Na_2SO_4 and nitrates.

The batch redox can be calculated as in Eq. (6):

$$Rx_h = \sum_{i=1}^{n} Rx_i M_i X_i \tag{6}$$

where, Rx_h is batch redox number, Rx_i is redox numbers of batch materials, M_i is quantity of batch materials for 2,000 kg sand, X_i is purity of batch materials.

In the present experimental study, the effect of redoxes condition and Fe_2O_3 content on the color

Table 1 **Sequence of metal ions in order of increasing ease of oxidation.**

Metal	Multivalent ion couples	
Chromium	Cr^{3+}	Cr^{6+}
Manganese	Mn^{2+}	Mn^{3+}
Cerium	Ce^{3+}	Ce^{4+}
Vanadium	V^{3+}	V^{5+}
Copper	Cu^{+}	Cu^{2+}
Arsenic	As^{3+}	As^{5+}
Antimony	Sb^{3+}	Sb^{5+}
Iron	Fe^{2+}	Fe^{3+}
Tin	Sn^{2+}	Sn^{4+}

Table 2 Redox numbers of batch materials (for 1 kg/2,000 kg sand).

Material	Redox number
Reducing	
Carbon (C)	-6.70
Iron sulphide (FeS)	-1.60
Pyrite (FeS_2)	-1.20
Carbosite (65% C)	-4.36
Arsenic (As_2O_3)	-0.93
Flowers of sulphur (25% S)	-1.10
Pure sulphur (S)	-4.40
Calcium fluoride (CaF_2)	-0.10
Oxidant	
Sodium sulphate (Na_2SO_4)	+0.70
Gypsum ($CaSO_4$)	+0.56
Barite ($BaSO_4$)	+0.40
Sodium nitrate ($NaNO_3$)	+0.32
Hematite (Fe_2O_3)	+0.25
Magnetite (Fe_3O_4)	+0.40
Manganese dioxide (MnO_2)	+1.09
Arsenic (As_2O_5)	+0.93
Potassium nitrate (KNO_3)	+0.27

formation of manganese containing soda-lime-silica glasses is investigated. For large interval of batch redox and at different level of iron and manganese oxides, color parameters (dominant wavelength, brightness and L-a-b coordinates) are examined.

2. Experiments

In order to obtain 100 g of glass, soda-lime-silica glass batches containing 0.12%, 0.2%, 0.4%, 0.6% and 0.8% pyrolusite by weight as MnO_2 source (99.95% MnO_2) were prepared with varying Fe_2O_3 contents and batch redoxes. In all batches, high purity sand with low iron content was used along with industrial grade soda, feldspar and limestone for the base glass composition. To increase the Fe_2O_3 content of the glass, hematite of 99.5% purity was used as the iron source. The batch redoxes were adjusted by adding anthracite (93.89% C) and sodium sulphate (99.83% $NaSO_4$) to the batches and were calculated as in Eq. (6) in the introduction.

After mixing the materials for 15 min in order to obtain homogeneity, the batches were melted in crucibles at 1,450 ^{o}C for exactly 3 h in an electrically heated furnace. Then, the melt was poured on a stainless steel plate and gradually cooled to room temperature in a chamber furnace for annealing. Finally, the glass samples were cut, grinded and polished to a thickness of 3 mm for optical transmittance measurements, which were carried out by using Perkin Elmer Lambda 950 UV/Vis/NIR (ultraviolet/visible/near infrared) spectrophotometer. The color differences in the glasses were observed and also chemical analyses of some of those were carried out by using Rigaku RIX 2000, XRF (X-ray fluorescence) spectrometer.

Fe_2O_3 and MnO_2 content and the batch redoxes of the total 19 sets of batches (68 samples) are given in Table 3. The final set of experiments was carried out with different types of manganese sources of different oxidation states of manganese, which are manganese nitrate of 98.5%, pyrolusite of 99.95% and potassium permanganate of 99% purity, having +2, +4 and +7 oxidation states, respectively.

3. Results and Discussion

In order to observe the effect of redox change on the absorption of Mn^{3+}, and therefore on the color of manganese containing glasses, Fe_2O_3 and MnO_2 contents were kept constant among the sets of comparison, while the batch redox levels were changed.

At 80 ppm Fe_2O_3 and 0.2 wt.% MnO_2 content, highest absorption of Mn^{3+} was obtained with +20 batch redox level. It can be observed that the intense purple color of the melted samples vanishes at +35 batch redox level, as seen in Fig. 3. To examine the effect of redox change at higher amounts of Fe_2O_3, the iron content of the glasses were increased to 0.020 wt.%, keeping MnO_2 at the same amount of 0.2 wt.%. Fig. 4 shows that the absorption maximum is reached at +25 batch redox level. Melting the glasses with the same amount of MnO_2, but even with a higher amount of Fe_2O_3 (0.050 wt.%) resulted in no color in the glasses.

Table 3 Theoretical Fe_2O_3 and MnO_2 content and the batch redoxes of the melted soda-lime-silica glasses.

Series #	Fe_2O_3 (wt.%)	MnO_2 (wt.%)	*Rx*
1.1	0.020	0.2	9
1.2	0.020	0.2	2
1.3	0.020	0.2	-2
1.4	0.020	0.2	-8
2.1	0.020	0.2	9
2.2	0.020	0.2	15
2.3	0.020	0.2	20
2.4	0.020	0.2	25
3.1	0.020	0.2	9
3.2	0.020	0.2	35
3.3	0.020	0.2	45
4.1	0.020	0.2	-2
4.2	0.020	0.2	9
4.3	0.020	0.2	20
4.4	0.020	0.2	25
5.1	0.008	0.2	20
5.2	0.020	0.2	20
5.3	0.050	0.2	20
5.4	0.100	0.2	20
6.1	0.008	0.2	-2
6.2	0.008	0.2	2
6.3	0.008	0.2	9
6.4 (5.1)	0.008	0.2	20
7.1	0.100	0.2	1.75
7.2	0.100	0.2	5.75
7.3	0.100	0.2	12.75
7.4 (5.4)	0.100	0.2	20
8.1 (7.3)	0.100	0.2	12.75
8.2 (5.4-7.4)	0.100	0.2	20
8.3	0.100	0.2	35
8.4	0.100	0.2	45
9.1 (5.3)	0.050	0.2	20
9.2	0.050	0.4	20
9.3	0.050	0.2	45
9.4	0.050	0.4	45
10.1	0.050	0.4	25
10.2	0.008	0.2	25
10.3	0.050	0.4	35
10.4	0.008	0.2	35
11.1 (9.2)	0.050	0.4	20
11.2 (10.1)	0.050	0.4	25
11.3 (10.3)	0.050	0.4	35
11.4 (9.4)	0.050	0.4	45
12.1	0.050	0.2	9
12.2	0.050	0.2	25
12.3	0.050	0.2	35
13.1	0.050	0.4	65
13.2	0.050	0.2	65
14.1	0.100	0.8	20
14.2	0.100	0.8	25
14.3	0.100	0.8	35
15.1	0.100	0.8	30
15.2	0.100	0.8	45
15.3	0.100	0.6	30
15.4	0.100	0.6	45
16.1	0.100	0.4	20
16.2	0.100	0.4	25
16.3	0.100	0.4	35
16.4	0.100	0.4	45
17.1	0.100	0.6	20
17.2 (15.4)	0.100	0.6	45
18.1	0.06	0.120	12
18.2	0.015	0.120	12
18.3	0.020	0.120	12
18.4	0.025	0.120	12
19.1 (manganese nitrate)	0.020	0.2	9
19.2 (pyrolusite)	0.020	0.2	9
19.3 (potassium permanganate)	0.020	0.2	9

When both Fe_2O_3 and MnO_2 contents were higher, 0.050% and 0.4% by weight, respectively, the absorption of Mn^{3+} increased as the batch redox was increased up to +35, but again the absorption of Mn^{3+} was lower at higher redox levels (Fig. 5).

Glass samples containing even higher amounts of Fe_2O_3 and MnO_2 are illustrated in Figs. 6 and 7, where Fe_2O_3 content is 0.100% by weight for both sets, but MnO_2 contents are 0.6 wt.% and 0.8 wt.%, respectively. Among the set containing 0.6 wt.% MnO_2, the highest absorption of Mn^{3+} was observed at +30 batch redox level, whereas the absorption maximum was at +35 batch redox level in the set containing 0.8 wt.% MnO_2.

The results of chemical and spectrometric analysis of the melted samples are given in Table 4, where theoretical (spectrometric) Mn^{3+} concentrations are calculated by Eq. (2) which is derived from Beer-Lambert law Eq. (1) mentioned in the introduction.

L-a-b coordinates of the melted samples according to their Mn^{3+} concentration are shown in Fig. 8, where

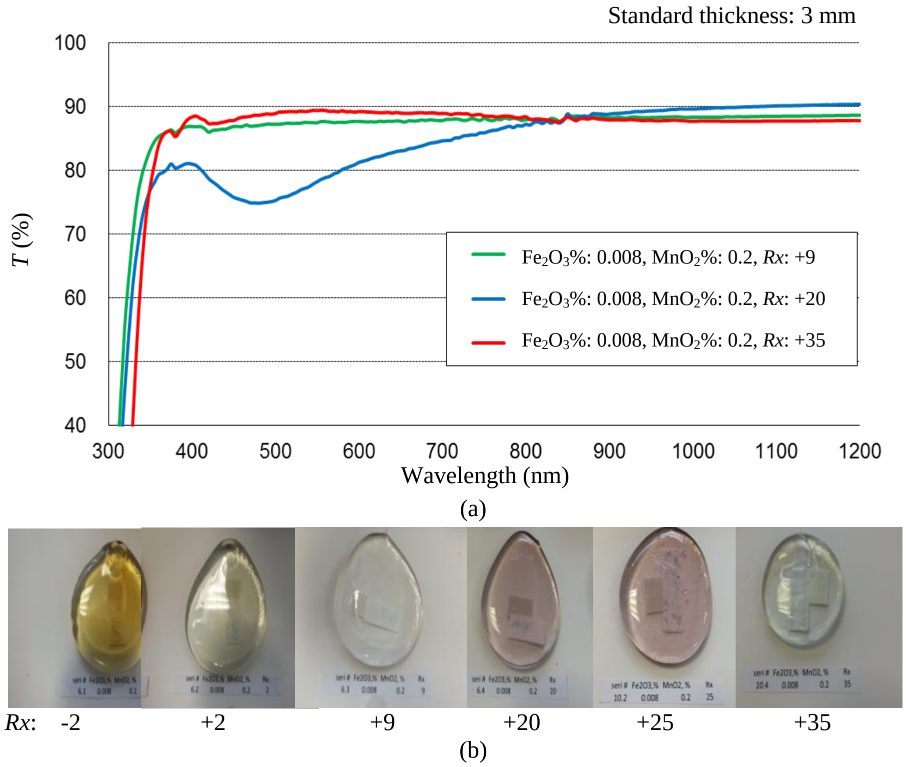


Fig. 3 Optical transmittance spectra (thickness: 3 mm) (a) and (b) colors of 0.008 wt.% Fe_2O_3 and 0.2 wt.% MnO_2 containing glasses at variable *Rx*.

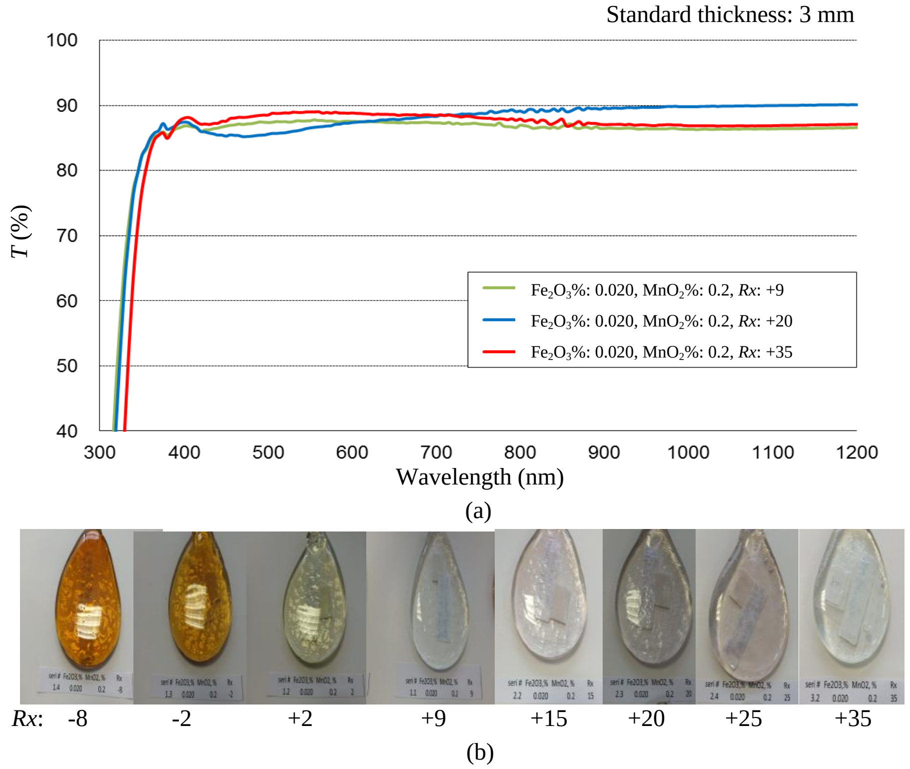
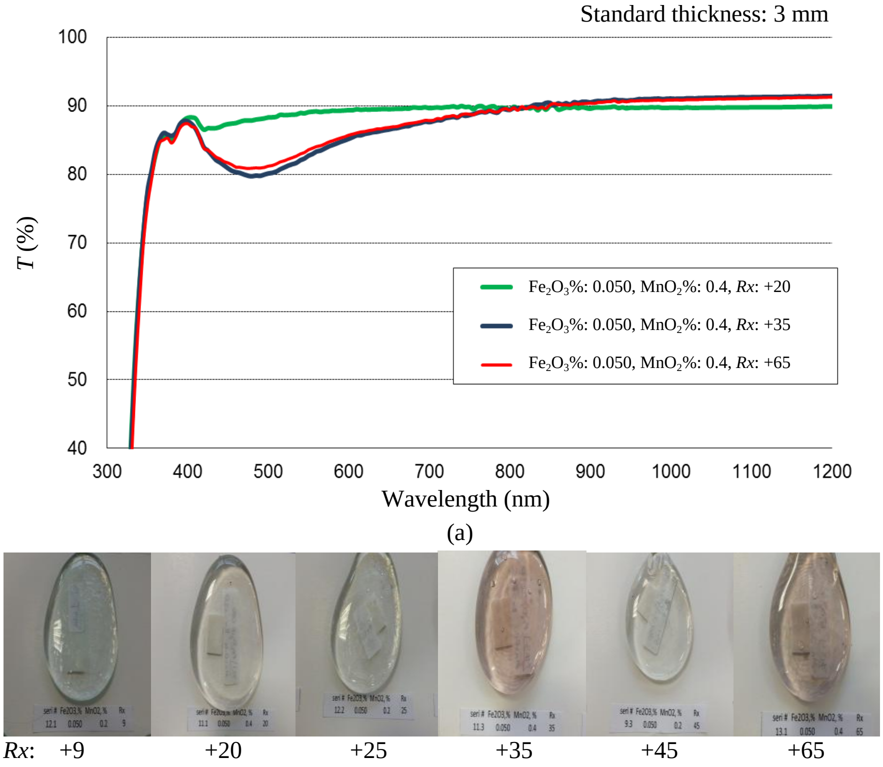


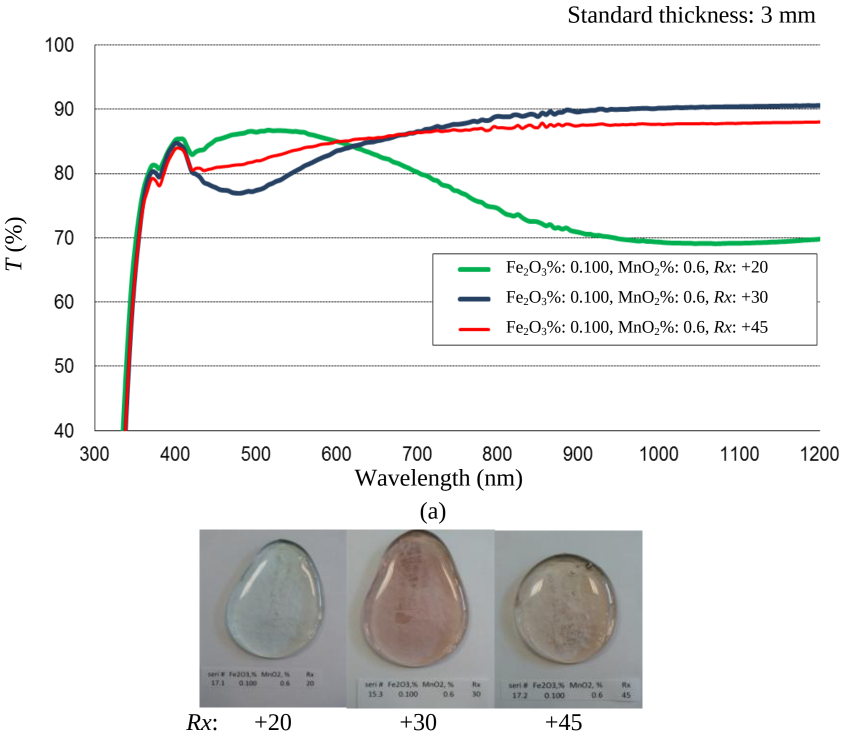
Fig. 4 Optical transmittance spectra (thickness: 3 mm) (a) and (b) the colors of 0.020 wt.% Fe_2O_3 and 0.2 wt.% MnO_2 containing glasses at variable *Rx*.



(a)

(b)

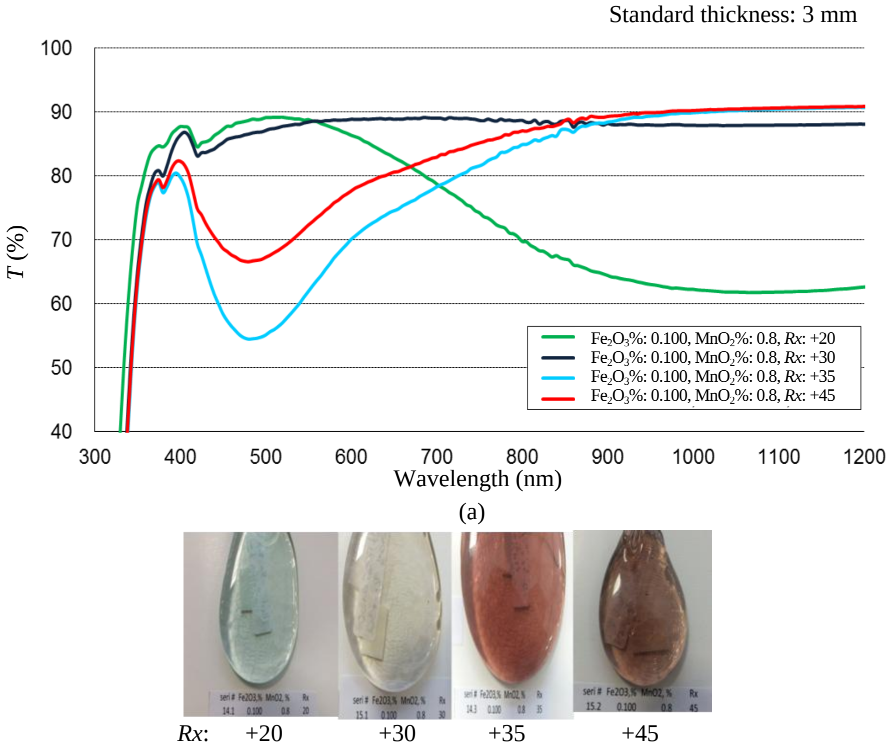
Fig. 5 Optical transmittance spectra (thickness: 3 mm) (a) and (b) colors of 0.050 wt.% Fe_2O_3 and 0.4 wt.% MnO_2 containing glasses at variable *Rx*.



(a)

(b)

Fig. 6 Optical transmittance spectra (thickness: 3 mm) (a) and (b) colors of 0.100 wt.% Fe_2O_3 and 0.6 wt.% MnO_2 containing glasses at variable *Rx*.



(a)

(b)

Fig. 7 Optical transmittance spectra (thickness: 3 mm) (a) and (b) colors of 0.100 wt.% Fe_2O_3 and 0.8 wt.% MnO_2 containing glasses at variable *Rx*.

Table 4 The results of chemical and spectrometric analysis of the melted samples (standard thickness: 3 mm).

Theoretical			Calculated from MnO analysis is of XRF		Spectrometric			
Fe_2O_3 (%)	MnO_2 (%)	Rx	MnO_2 (%)	Mn (%)	Brightness (%)	T (490 nm)	Mn^{3+} (%)	Dominant wavelength
0.008	0.2	9	0.225	0.143	87.5	87.4	0.019	575
0.008	0.2	20	0.238	0.150	78.6	75.3	0.074	590
0.008	0.2	35	0.240	0.152	89.2	88.9	0.013	570
0.020	0.2	9	0.239	0.151	87.5	87.5	0.018	575
0.020	0.2	20	0.235	0.149	86.6	85.5	0.027	585
0.020	0.2	35	0.239	0.151	88.8	88.5	0.014	570
0.050	0.4	20	0.475	0.301	89	88.2	0.016	575
0.050	0.4	35	0.491	0.311	83	80.5	0.051	586
0.050	0.4	65	0.493	0.311	83.8	81.3	0.046	586
0.100	0.6	20	0.703	0.445	85.9	86.8	0.022	520
0.100	0.6	30	0.721	0.455	80.9	77.7	0.063	590
0.100	0.6	45	0.716	0.452	83.7	82.5	0.043	580
0.100	0.8	20	0.984	0.622	87.8	88.9	0.013	500
0.100	0.8	30	0.966	0.610	88.2	87.1	0.021	575
0.100	0.8	35	0.951	0.601	63.4	55.3	0.188	606
0.100	0.8	45	0.963	0.609	73.1	67.4	0.115	594

colored glasses have higher Mn^{3+} concentration. It should be noted that, as Mn^{3+} concentration increases, not only the color gets more intense, but also the dominant wavelength changes.

For the purpose of examining the effect of Fe_2O_3 amount in manganese containing glasses, batches containing 0.120 wt.% MnO_2 were melted at a constant batch redox level of +12, with variable amounts of

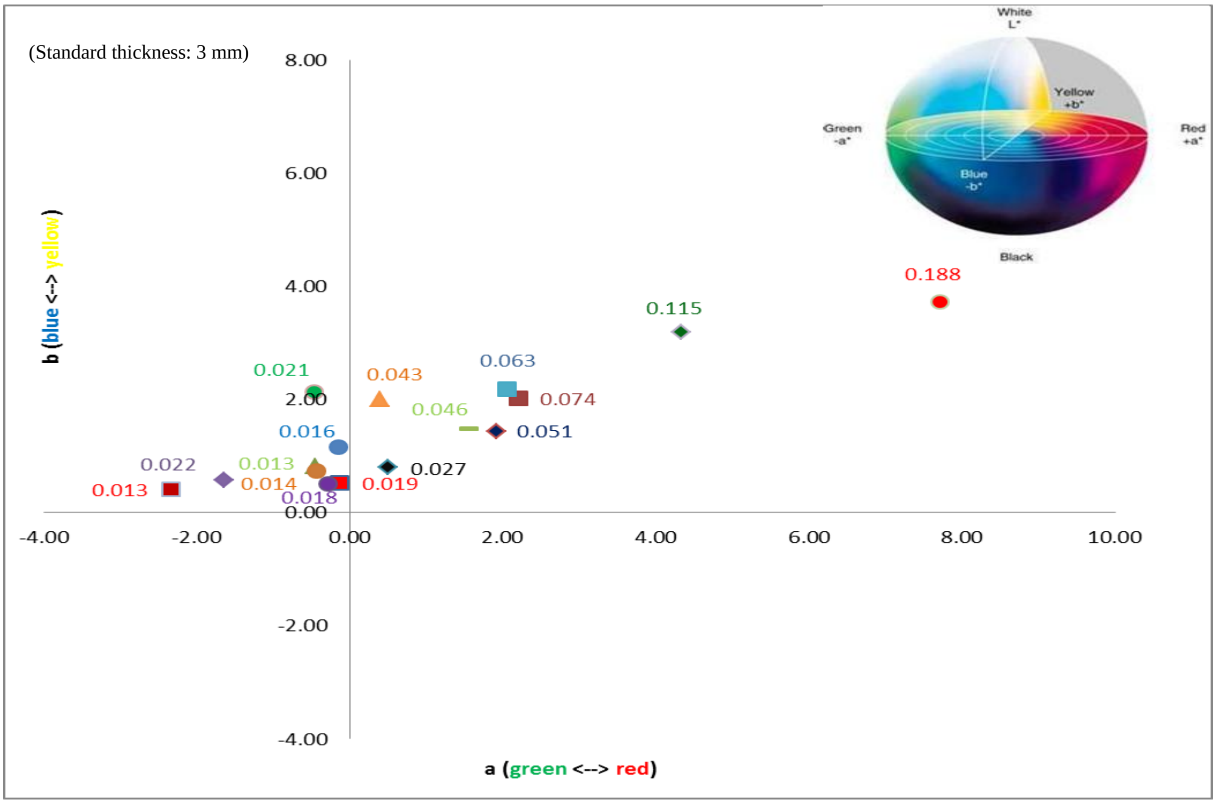


Fig. 8 L-a-b coordinates of the melted samples according to Mn^{3+} concentration (standard thickness: 3 mm).

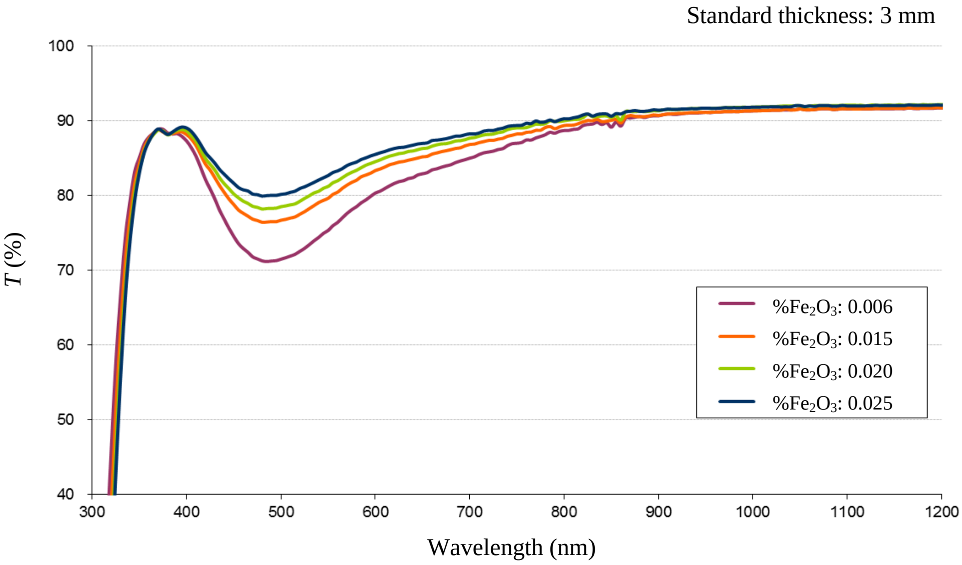


Fig. 9 Optical transmittance spectra (thickness: 3 mm) of 0.120 wt.% MnO_2 containing glasses at +12 batch redox with variable Fe_2O_3 content.

Fe_2O_3 (0.06%, 0.015%, 0.020% and 0.025% by weight). In Fig. 9, it can be observed that as Fe_2O_3 amount increases, the absorption of Mn^{3+} decreases.

As a final set of melting experiments, different sources of manganese with different oxidation states were compared. Batches all of which contain 0.2 wt.% manganese as MnO_2, but from manganese nitrate, pyrolusite and potassium permanganate, respectively, were melted with a content of 0.020 wt.% Fe_2O_3, at a batch redox of 9. All of the samples were colorless,

Table 5 The results of spectrometric analysis of the melted samples with different manganese sources samples (standard thickness: 3 mm).

Manganese sources	Manganese nitrate	Pyrolusite	Potassium permanganate
Sample thickness (mm)	2.87	2.81	2.88
Brightness (%)	90.5	90.6	90.8
T (490 nm)	90.3	90.4	90.6
Mn^{3+} (spectrometric)	0.0070	0.0069	0.0058
Dominant wavelength	575	575	575

having very close dominant wavelengths. The results of spectrometric analysis of the melted samples are listed in Table 5.

4. Conclusions

The effect of batch redox condition and Fe_2O_3 content in soda-lime-silica glasses containing various amounts of MnO_2 has been investigated by multiple melting experiments.

As a result of above mentioned experiments, it is concluded that, as manganese content is increased at constant amount of Fe_2O_3 and constant batch redox, manganese absorption increases, therefore, the amethyst color deepens from colorless to dark purple.

When the content of Fe_2O_3 is increased at constant amount of MnO_2 and batch redox, the color fades and therefore more MnO_2 is required to achieve the color, i.e., higher oxidation levels are needed to have Mn^{3+} absorption.

However, above some level of oxidation as batch redox, Mn^{3+} absorption decreases depending on the Fe_2O_3 content. At moderate oxidation levels, higher absorption of Mn^{3+} is achieved.

As a final remark, it is necessary to mention that new methods are needed to be established in order to determine if different oxidation states of manganese, which do not give amethyst color, are present in the glass at high oxidation levels.

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