Global Scientific JOURNALS

GSJ: Volume 11, Issue 3, March 2023, Online: ISSN 2320-9186
www.globalscientificjournal.com

EFFECTS OF TRANSITION METAL ADDITIVES ON THE PHYSICOCHEMICAL PROPERTIES OF SOME SELECTED GLASS

***Olaoluwa Ayobami Ogunkunle[1], Saheed Ajekayode Adedokun[1], Sunday Emmanuel Olajide[1] and Oluwamumiyo Dorcas Adeojo[2]**

[1]Department of Chemistry, Obafemi Awolowo University, Ile-Ife 220005, Nigeria
[2]Department of Chemistry, University of Ibadan, Nigeria

*Corresponding author
Email address – oogunkunle@oauife.edu.ng
Phone number - +2349038099962

Abstract

This research work focused on the production of three coloured glasses namely; Amber, Green and Flint, assessment of the impact of various ratios of transition metal additives in glass, characterization of produced glass samples and ascertaining the level or existence of additional bonds formed. Comparisons were also made between normally produced glass and those infused with transition metal additives where the colour, intensities, bonding, and strength were studied.

Keywords: amber, green, flint glass

1. INTRODUCTION

Glass is an inorganic product of fusion which has been cooled to a rigid condition without crystallizing (Begriffe et al, 1986). Glass refers to a hard brittle transparent amorphous solid, such as that used for windows, bottles, eye wear just to mention a few. It is amorphous because it is neither a solid nor liquid but exists in a vitreous or glassy state.

Many glasses contain silica as their main component and glass former. Glass is normally lustrous and transparent in appearance and it shows great durability under exposure to the natural elements (Horst, 1991). These three properties namely lustre, transparency and durability make glass a favoured material for household objects such as window panes, bottles, and light bulbs. However, neither any of these properties alone nor all of them together are sufficient for a complete description of glass (Horst, 1991). Transition metals have recently being used in glass production as additives and this is due to their ability to form complexes, that is they have the ability to form multiple bonds thereby making glass very stable. Glass plays an essential roles in various scientific and industries fields. The optical and physical properties of glass make it suitable for application such as flat glass, container glass, optoelectronic materials, Laboratory equipment, thermal insulators (glass wool) reinforcement fibers (glass-reinforced plastic, glass fiber, reinforced concrete) and art (Horst, 1991). The broad classes of glass include; sodalime, borosilicate and lead

glasses. The classes based on uses include; tinted, low emission, x ray protection, chemically strengthened, sand blasted and fused silica glasses. Additives used for fining in glass during production are $NaNO_3$, AS_2O_3, NaCl and Na_2SO_4 are added to remove bubbles in the glass. The fining agents decompose, given off large bubbles of gases in the melt which swept out the small bubbles that are always formed. Decolorizers such as MgO which precipitate out Iron and Sulphur can also be added. Soda ash works as a flux in the batch and it is added with metals to facilitate its fusion and to render colour fusible in the glass. Sodium carbonate is also known as the soda ash in the industry and its function as a flux in the batch by lowering the melting point of the glass melt to about 1500 ^{0}C (Manning and Diken, 1975). Pigments used in impacting colour in these glasses are; for flint glass, cobalt and selenium are being used but antimony oxides and tin compounds can also be used. For green glass, iron chromite is being used, but iron oxide can also be used. For amber glass, coal and iron oxide are used. But manganese oxides, carbon oxides and sulfur compounds can also be used (David, 2006 and Barkhau, 1988).

Flint glass composition are employed for enclosing food production in applications in which it is desirable to be able to view the product through the container wall. They are commonly used for carbonated drinks such as Coca-cola, Fanta, Pepsi to mention a few. Flint glass is used as soft drink bottles for which transmission of light through the glass does not affect the flavour of the product.

Amber glass is employed for products such as alcoholic beverages based on hops, so that the absorption properties of the glass decrease transmission of light wavelength through the containers wall which might potentially degrade the products e.g. Gulder, Star, e.t.c. Amber glass is used as Beer bottle because light strikes affect the resultant flavour or aroma of the Beer. So, to minimize this effect beer has been bottled in amber glass which reduces the transmission of light at the

wavelength where the chemical reaction occurs. The colour of the amber glass is the best colour for beer bottles because it offers the best protection against light.

Green glass is employed for product for which transmission is not so significant in affecting the product such as soft drink bottle e.g. star, sprite but thickness of green glass is very important to minimize light transmission (Dillon, 1958 and Horst, 1991). Green glass is used as soft drink bottles for which transmission of light through the glass has to be at minimal level (IOS, 1985). It was also found that flint and amber glass are reactive with each other due at least in part to differing oxidation states of these glasses. It is thus necessary when converting flint glass to amber glass container to fully remove any residual flint glass in the furnace and further equipment through redox reaction within the amber glass (Simpson and Myers, 1978).

2. EXPERIMENTAL

All the reagents used were of analytical grades. The raw materials were subjected to purity test to ascertain if they were appropriate for use and some of the tests carried out were percentage purity of MgO, CaO, and Fe_2O_3.

2.1 Treatment of Raw materials

Raw materials used for production were collected from Batch and Furnace Department of Beta glass Nigeria Plc, Agbara industrial estate, Agbara. The different starting raw materials were subjected to drying in an Oven at a temperature of about 1000 ^{0}C for about 30minutes before cooling and grinding using different mortars and pestles. These starting raw materials include: Silica sand (treated), Soda ash, Limestone, Feldspar, Sodium sulphate, Ferric oxide, Iron chromite, Coal, Selenium, Cobalt, Amber cullet (Ground), Green cullet (Ground), Flint cullet (Ground). Anhydrous Iron chromite, ferric oxide, coal, selenium, cobalt and sodium sulphate in the required

grain size 0f 0.12-0.15 mm were used for this study (Alexander, 2009). Information on the batch formulation for the commercial production of these glasses was obtained from the Batch and Furnace Department and these were then reduced to laboratory scale.

2.2 Procedure

The raw materials (batch) for the glass to be produced were weighed into a clean 250ml beaker using mettle Toledo scale (Analytical balance) and mixed thoroughly using spatula for about 5 minutes for homogenization of the batch. The batch of 30g was poured into the 25ml platinum dish and then transferred into the muffle furnace which has been pre-heated to about 500 ^{0}C using tongs. The temperature of the muffle furnace was then increased by 100 ^{0}C in every 10 minutes until the temperature of the muffle furnace attained 1400 ^{0}C. At this temperature, the molten glass will be formed (Alexander, 2009). The red hot platinum dish containing the molten glass was then removed from the muffle furnace with the aid of heat resistant gloves and tong. Spatula was used to pour the molten glass into the mould which has been lined by glass wool. The mould containing the formed glass was then further covered with glass wool to allow the annealing operation to take place, preventing stress in the glass (Horst, 1991). The solid glass was removed after about one hour and kept in small plastic container and labeled. The platinum dish was then put back into muffle furnace to re-melt the remaining glass in the platinum dish. When the glass has melted which takes about 10 minutes. It was then removed with tong and the molten glass was poured on the mould but it was not covered with the fiber wool. The glass obtained was used for further analysis such as determination of percentage CaO, MgO, and Fe2O3. The remaining glass in the platinum dish solidified in it and the platinum dish was then put in a small plastic container, containing a solution of hydrochloric acid for about 30 minutes. After which, it is removed and washed with water with aid of Small stainless steel knife to remove the dissolved glass from the

platinum dish. The platinum dish was then put on the sand bath which has been preheated and hydrochloric acid solution was poured on it. These operations (heating and washing) were repeated for three times for all the glasses in the platinum dish to be removed.

2.3 Formulation of the different types of Glass produced

The same method of production was used in the production of the glass with a variation in the amount of normal additives used in commercial production and the transition metals additives added to the production mixture. The variations include: Normal Glass, Normal Glass + one transition metal additive, Normal glass + Two transition metal additives. The additives did not exceed the stipulated amount and were in the ratio 1:0, 1:1, 1:3 of Normal: Transition metals additive(s). The three types of the glass samples were also produced using no additive (0:0)

2.4 Characterization methods

Infrared analysis

The Fourier transform infrared spectroscopy (FTIR) was carried out on the glass samples produced at the Nigeria Institute of Science and Laboratory Technology (NISLT) located in Samonda, Ibadan using the Perkin Elmer FT-IR spectrum BX. The results obtained are presented in Table 4.1.

Ultraviolet analysis

The Ultraviolet spectroscopy was carried out on the produced samples in Chemical Research Laboratory in Ladoke Akintola University of Technology, Ogbomoso using the Genesys 10 UV-VIS Spectrophotometer model No 335900 and the results obtained were presented in Tables 4.2 a & b. This analysis was carried out for both the Near and Far UV region of the spectrum.

Colour transmittance

The colour transmittance of the glass samples produced was carried out using UV- VIS Spectrophotometer model No CE 2041 and the colour transmittance determination was carried out by cutting the glass samples into a shape that can enter the hole where the samples are kept for the analysis using Glass cutting machine. After which the sample colour transmittance was determined by setting the spectrophotometer to the appropriate wavelength for the different glass types. For Amber coloured glass, the wavelength is 550nm, for Green, it is 520nm while that of flint is 510nm. The glass sample was then put to the spectrophotometer and its colour transmittance was determined using the relation:

$$T_C = Log^{-1} (T_m/92 \times t_c/t_m + \log 92)$$

Where T_C = Calculated glass sample Transmittance

T_m = Measured transmittance

t_c = Corrected thickness=2mm

t_m = measured thickness

Specific Gravity

The specific gravity of the samples was determined using specific gravity machine Model No DC. It was determined by cutting a small lump of the glass in square shape and dropped into the machine containing a standard lump and the dropping temperatures of the two lumps were noted. The specific gravity of the glass samples were then determined using the relation:

$$S.G = 0.00018\ \Delta \pm 2.5025$$

Where Δ is the difference in the dropping temperature of the standard and the glass lump. 0.00018 is a constant. 2.5025 is added if the lump drops before the standard and subtracted if it drops after the standard.

2.5 Determination of the percentages of Iron, Magnesium and Calcium oxides in the glass samples

Samples were digested using platinum dish and combination of concentrated hydrochloric acid and water (1:1) on a sand bath until a clear solution was obtained. This was now diluted to the mark with distilled water in a 25ml standard flask. This served as the stock solution.

For Fe_2O_3:

Tartaric acid (10%) was added to the stock solution, after which 2-3 drops of p-nitrophenol was added followed by the addition of (1:1) NH4OH with continous shaking until a colour changed to yellow. A solution of (1:1) HCl was added until the solution decolourized. 5ml of 10% Hydroxylamine hydrochloride solution was added, followed by the addition of 10ml of o-phenanthroline solution. The solution was then made up to 100ml mark with distil water with rigorous mixing. The absorbance of the resultant solution was taken using spectrophotometer and compared with that of the blank.

For CaO:

To 5ml of the stock solution, 25ml of 0.2M NaOH was added and a pinch of murexide indicator. The resulting solution was immediately titrated against 0.01M EDTA until the colour changed from pink to purple. The titre value was then used in the estimation.

For MgO:

5ml of the stock solution was pipetted into a clean 250ml beaker before 25ml of pH 10 Buffer solution and a pinch of solochrome Black T were added. The resulting was then titrated against 0.01M EDTA until the colour changed from wine red to blue. The titre value was then used in the estimation.

3. RESULTS AND DISCUSSION

3.1 Infrared spectroscopy of the glass samples

The relevant infrared spectra were studied by the comparison of the bands obtained from the spectrum of the flint glass containing no additives with other glass samples containing different concentrations and ratios of transition metal additives.

The absorption bands in the region 3900-3400cm^{-1} are broad and are assigned to stretching vibrations of Alcoholic -OH, Phenolic -OH, Carboxylic acid -OH, and –NH of Amine. With alcoholic -OH band appearing between 3700-3600cm^{-1}, aromatic -OH band appearing between 3665-3600cm^{-1}, Carboxylic acid -OH band appearing between 3565-3455cm^{-1} and the Amine -NH appearing between 3465-3265cm^{-1} (Wade, 1995). All these bands are broad and this may be due to the different vibrational modes of these functional groups (Ecarhos and Walrafen, 1986). It was observed that the band due to alcoholic -OH and the carboxylic -OH were shifted to lower wave number in all the spectra compared to the standard and also it does not appear in some of the spectra. Olefinic C=C and Aromatic C=C appeared between 1660-1610cm^{-1} and between 1565-1500cm^{-1}respectively and the bands due to these two functional groups appear to the lower wave number for the standard compared to the spectra of all other transition metal dopped glass samples (Wong and Angel 1976). Other bands found in this region of the spectrum, are due to functional groups such as aryl ether -CO stretching vibration which appear between 1350-1255cm^{-1} but it

does not appear in the standard, amine C-N bands which appear between 2400-2200cm^{-1}, and it is lower for the standard than other glass samples except for amber glass with $Mn^{2+,}$ and amber glass with Fe^{2+} and Mn^{2+} in the ratios (1:1) and (1:3). The band due to aromatic C-H bending vibration which appears between 900-800cm-1was observed to appear to the lower wave number for the standard than all other transition metal dopped glass samples except for green glass sample with Co and Ni chloride (1:1) (Merzbacher and White 1991). All the bands due to these functional groups are broad and may be due to organic compounds whose possible source may be from the use of coal in the production of amber glass. The presence of the above mentioned organic functional groups in all the spectra of the transition metal dopped glass samples may be due to the use of coal in the production amber glass sample and the reason being that, all the glass samples were produced in the same medium which is the platinium dish (Klaus, 2003). The same platinium dish was used to produce all the glass samples and after each production, it is dissolved with dilute HCl but not all the glass remaining in the platinium dish was able to be removed completely before it was being used for further production. So there may be interference of these functional groups present in the coal with all other glass sample being produced (Alexander, 2009).

In the fingerprint region, the band due to Si-O stretching vibration, appeared between 1066-1016cm^{-1}, the standard showed only one broad band for Si-O vibration, but for the spectra of all other transition metal dopped glass samples, they show multiplet bands but these multiplet bands are not sharp in all the spectra which may be due to different vibrational modes of Si-O in the spectra of the glass samples with transition metal (s) (Ecarhos and Walrafen, 1986), The band due to Si-C stretching vibration appeared between 780-750cm^{-1}. For the standard, it appeared at 780cm^{-1} but it is lower in the spectra of all other glass sample. The band due to Si-C vibration in the spectra of transition metal dopped glass samples except the standard showed multiplet bands. Also,

the band due to C-S stretching vibration appeared between 700-630cm^{-1} and it showed multiplet bands for the spectra of all other glass samples except for the standard (Karlsson, et al 1989, Wade, 1995),

The multiplet bands in the fingerprint region which are very sharp with high intensities, may due to the transition metal (s) in the dopped glass samples and since these multiplet bands were not found in the spectrum of the standard, so this may be an indication that bonding has taken place, and this may also be an indication of an extensive bonding in the dopped glass samples (Ecarhos, et al 1986). Also, this observed multiplet bands in the fingerprint region may also be an indication of an increase in tensile strength of the dopped glass samples.

It was observed that amber glass with Fe^{2+} and Mn^{2+} showed multiplet bands in the fingerprint region for ratio 1: 3 while the one with ratio 1:1did not. Also, green glass with Ni^{2+} and Mn^{2+} and amber glass with Mn^{2+} showed multiplet bands in the fingerprint region. It was also found that all the bands in all the region s of the spectra of amber glass with Ni^{2+} were not all that sharp. It can then be said that Ni and Mn chloride salts are not a very good glass 2274opant compared to Co and Fe chloride but the spectrum of glass sample dopped with Mn showed sharper band than that dopped with Ni but if Mn and Ni salts are used together as double salt in higher proportion, they are very good glass doppants (Dunken and Doromus 1987). In all the cases, bands tentatively assigned to the bending vibrations of aromatic C-H were present. Likewise phenolic, -OH, alcoholic -OH, carboxylic acid -OH, amine -NH, -CN, C-CH3 were found to be present in all the spectra. M-O bands were present in the fingerprint region of the spectra of the transition metal dopped glass samples except the standard. The lower shifts have often been used as an indication of increase in electron density on the metal ion and the strength of the bond in the glass sample (Minami 1995). Bonding with the metal ions present in the glass samples could be through the,

phenolic, -OH, aromatic -OH, carboxylic -OH and aryl ether -CO present within the glass system (Minami, 1987)

3.2 UV Spectroscopy of the Glass Samples

The relevant UV spectra were studied by comparing the spectrum of the standard with the spectra of transition metal doped glass samples in the near UV region (190 400nm) and far UV region (400-1100nm) of the spectrum.

It was observed that, the UV spectrum of the standard showed maximum absorbance peak 0.205 at λ max = 232nm while the spectra of other glass samples showed higher absorbance peak as high as 1.154 with at λ max =234nm for flint glass with Co^{2+} (1:1) and also the absorbance peak for other glass sample are higher than the standard in the near UV region. This implies that, there is a lower chemical shift in the spectra of the standard compared to the spectra of other glass samples (Abdel and Morsi, 1997). This may be an indication that, reaction has taken place. The higher absorbance peaks observed for all other glass sample than the standard, could be as a result of the change in the chromophore which are the transition metal ions through photo ionization (Morsi and Nassar, 1978)

For the far UV region (400-1100nm), it was observed that the absorbance peak 0.271 at λ max =985nm, for the standard is higher than that of other glass samples (table 2b), except for green glass with Ni^{2+} and Co^{2+} (1:1), amber glass with Ni^{2+} (1:1) and amber glass with Fe^{2+} & Mn^{2+} (1:3) which have absorbance peaks higher than that of the standard. It was also observed that absorbance peaks of the UV spectra of all the glass samples are higher in the near UV region than in the far UV region except for the standard whose absorbance peak is higher in the far UV region (0.271, λ max=985nm) than in the near UV region (0.205, λ max=217nm) and also, the absorbance peaks

of the spectra of all the glass samples appear in different position in the near UV region but they appear very close to one another in the far UV region (Griscom, 1990). It was also observed that, in the UV spectra of some of the glasses, some absorbance peaks are very close to each other and even the same in some cases in both near UV region and far UV region.

In all the cases, absorbance peaks were observed for all the glass samples in both near UV region and the far UV region and these absorbance peaks are as a result of formation of ion of the transition metal by photo-ionization of these transition metals through irradiation with light (Moncke and Ehrt, 2004). This is shown in the equations below:

(i) $Mn^{2+} + hv \rightarrow Mn^{3+} + e^-$

(ii) $Fe^{2+} + hv \rightarrow Fe^{3+} + e^-$

(iii) $Co^{2+} + hv \rightarrow Co^{3+} + e^-$

(iv) $Ni^{2+} + hv \rightarrow Ni^{3+} + e^-$

So, the absorbance peaks in the spectra of the dopped glass samples are due to ionization of the ion of the transition metal but the absorption takes place at different wavelength for each of the transition metal dopped glass samples in both near UV and far UV region (Williams et al, 1986). For instance, the spectrum of the green glass doped with Co^{2+} showed three peaks at 214, 232, and 364nm in the near UV region but it gives four peaks at 433, 418, 457, and 481nm and the peaks are very close to one another in the far UV region (Weyl, 1959). Also the Ni^{2+} in amber glass sample can undergoes photochemical reaction giving two peaks at 232 and 364nm in the near UV region but it gives four peaks very close to one another at 991, 1003, 1039, and 1093nm in the far UV region (Natura and Ehrt, 1995). The Mn^{2+} can also undergo photo oxidation giving four peaks

in the spectrum of amber glass sample at 205, 217, 238, and 346nm in the near UV region, and also given four peaks very close to one another at 1027, 1039,1060,and 1078nm in the far UV region (Bates and Mackenzie, 1962). Fe^{2+} can also undergo photo oxidation, for example, in amber glass sample, it gave three peaks at 196, 238, and 370nm in the near UV region but it gives four peaks at 994, 1006, 1015, and 1039nm which are very close to one another in the far UV region (Ehrt and Seeber 1993). But it was observed that, there is lower chemical shift for the standard compared to all other glass samples with transition metal ion and this may be an indication that bonding has taken place which could be more for the glass dopped with transition metals than the standard

3.3 Colour Transmittance of the Glass Samples

All the colour transmittance obtained were appropriate in bottling or storing of items because they fell within the range stipulated for Green, Amber and Flint glasses which makes them appropriate for use for different purposes except for Green glass Ni and Mn, Amber glass with Co, Green glass with Ni, Co and Green glass with no additive whose values were lower than the standard thus, having limited uses in terms of storage (Bamford, 1977). The corresponding decrease in colour transmittance was due to the presence of Ni and Mn. This can also attributed to the size and extent of linkage between the normal bonds and the added transition metal additives (Weyl, 1999). The table shows the colour transmittance of different glass samples below

Samples	Colour transmittance (%)
Normal Green glass	74.49

Normal Amber glass	44.29
Normal Flint glass	42.00
Green glass with $Ni^{2+}$and Mn^{2+} (1:1)	49.73
Amber glass with Fe^{2+} and Mn^{2+} (1:1)	74.46
Amber glass with Mn^{2+} (1:1)	64.14
Amber glass with Ni^{2+} (1:1)	40.90
Amber glass with Co^{2+} (1:1)	30.65
Green glass with Ni^{2+}(1:1)	41.84
Green glass Co^{2+} (1:1)	60.49
Amber glass with Fe^{2+} (1:1)	71.27
Green glass with Ni^{2+} and Mn^{2+}(1:3)	46.16
Amber glass with Fe^{2+} and Mn^{2+}	63.71
Amber glass with no amber additives	55.34
Green glass with no green additives	66.48
Flint glass with no flint additives	61.32

3.4 Specific gravity of the Glass Samples

The result of the specific gravity test of the produced glass samples as presented in table below shows that, all the glass samples had values that conform with the standard density (2.45-2.54gcm^{-3}) for a soda-lime glass. This is an indication that, they are all very stable and inert to environmental and atmospheric conditions. It must be noted that, values lower than this, makes glass water soluble (shelty, 1997).

Samples	**Δ (^{0}C)**	**Specific Gravity (gcm^{-3})**
Normal amber glass	1.3	2.5053
Normal green glass	6.0	2.4968
Normal flint glass	1.4	2.5051
Green glass with Ni^{2+} and Mn^{2+} (1:1)	3.3	2.5017
Amber glass with Fe^{2+} and Mn^{2+} (1:1)	2.6	2.5029
Amber glass with Fe^{2+} (1:1)	3.8	2.5008
Green glass Co^{2+} (1:1)	2.4	2.5033
Green glass with Ni^{2+} (1:1)	2.0	2.5040
Amber glass with Mn^{2+} (1:1)	2.6	2.5029
Amber glass with Ni^{2+} (1:1)	4.5	2.4995

Amber glass with Co^{2+} (1:1)	1.6	2.5047
Green glass with Ni^{2+} and Mn^{2+} (1:3)	3.4	2.5015
Amber glass with Fe^{2+} and Mn^{2+} (1:3)	3.1	2.5020
Amber glass with no additives	0.9	2.5060
Green glass with no additives	1.8	2.5044
Flint glass with no additives	0.6	2.5065

3.5 Metallic Oxides Composition of the Glass Samples

The results of the metallic composition of the various glass samples showed that, the raw materials used for their production are pure and suitable for this research.

Samples	**Fe_2O_3**	**MgO**	**CaO**
Normal amber glass	0.0365	1.61	10.93
Normal green glass	0.0928	1.41	11.49
Normal flint glass	0.00938	1.81	11.21
Green glass with Ni^{2+} and Mn^{2+}(1:1)	0.276	1.61	11.49

Amber glass Fe^{2+} and Mn^{2+} (1:1)	0.242	2.01	10.65
Amber glass with Fe^{2+} (1:1)	0.041	1.61	10.93
Green glass Co^{2+} (1:1)	0.103	1.61	11.77
Green glass with Ni^{2+} (1:1)	0.264	1.81	10.65
Amber glass with Mn^{2+} (1:1)	0.116	2.42	10.35
Amber glass with Ni^{2+}(1:1)	0.117	2.42	10.65
Amber glass with Co^{2+} (1:1)	0.119	2.22	10.93
Green glass with Ni^{2+} and Mn^{2+} (1:3)	0.257	1.21	11.77
Amber glass with Fe^{2+} and Mn^{2+}(1:3)	0.274	1.81	10.93
Flint glass with Co^{2+} (0:1)	0.001	1.12	10.45
Amber glass with no additives	0.0302	1.61	11.21
Green glass with no additives	0.103	1.81	11.49
Flint glass with no additives	0.0036	2.01	10.93

4. CONCLUSION

From this study, it can be concluded that transition metal additives have extensive effect on the physico-chemical properties of glass. The IR showed that more bonds were formed within the

glass structure by the addition of transition metal additives through Alcoholic –OH, Phenolic –OH, Carboxylic –OH, due to the lowering of the absorption bands (increase in electron density), and the appearance of multiplet peaks and emergence of new bands. Ultraviolet spectroscopy also indicated that, more bonding have taken place in the glass dopped with transition metal additives. The stability of the glass sample produced was buttressed by the specific gravity result which indicated that, they can not degenerate by various conditions. Thus, making them appropriate as materials for storage. Some of the mixed ratios of the doppants gave very good colour transmittance for relatively small size transition metal (due to high level of networking) while large size ions gave low networking and low bond formation). In conclusion, it can be said that transition metal additives enhance the appearance and durability of glass, most especially when they are in appropriate concentration. Double transition metals additives gave better result.

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