Artificial neural network analysis of optical measurements of glasses based on Sb_2O_3

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In the paper we present application of artificial neural network (ANN) on relation between glass composition versus optical transmittance of the chosen glass systems of Sb_2O_3 - $PbCl_2$ and Sb_2O_3 – PbO – M_2O, where M was Na, K and Li, respectively. The excellent prediction ability of special ANN program developed for this study demonstrates the possibility to influence the glass composition to obtain asked optical properties. The measurements of the temperature dependencies of the direct electric conductivity show the strong influence of the concentration of the individual glass compounds of systems Sb_2O_3 - $PbCl_2$ and Sb_2O_3 – PbO – M_2O (M is Na, K, Li) on their electric and dielectric properties. Glasses own the same mechanism of the electric conductivity with activation energy, which goes to the value 3.75 eV when temperature is higher than 250 °C.

Similarly optical transmittance T of systems Sb_2O_3 - $PbCl_2$ and Sb_2O_3 – PbO – M_2O strongly depends on the glass composition and the amount of defects, too. The glass $70Sb_2O_3$ – $30PbCl_2$ reached the highest value of T. The minimal content of defects in its volume makes these glasses very perspective for next searching.

The measurements of the complex modulus M^* of mentioned glasses showed their high sensitivity to the changes of glass structure connected with the creation of different sort and the amount of defects. The sensibility of the used methods is comparable with the usual exploited methods (X-ray analysis, optical microscopy) and makes possible to assess partially the quantitative occurrence of defects in the glass volume.

A model of neural network for prediction of the optical transmittance was created. Model enables to predict the transmittance with sufficiently small error. After evaluation of results we can state that exploitation of neural networks is advantageous, if it is necessary to express complex mutual relations among sensor-based data. Neural networks are able to realize and appropriately express general properties of data and relations among them and on the contrary to suppress relationships which occur sporadically or they are not sufficiently reliable and strong. Their usage enables retrieval of relationships among parameters of the process which with use of common methods are not possible to trace for reason of their mutual interactions, big amount and dynamics. Use of a neural network seems to be suitable tool for estimating different important optical parameters.

(Received November 05, 2015; accepted April 05, 2016)

Keywords: Heavy metal oxides glasses, Artificial neural networks, Transmittance, Dielectric properties

1. Introduction

Optical materials suitable for utilizing in the infrared region determined for the radiation transport in various lasers are under intensive research for a long period. The attention is devoted to the active and passive optical fibres prepared from suitable glasses based on the heavy metal oxides. Although the most of research interests is directed to telluride glasses there is possibility for glasses based on the antimony oxide to achieve similar physical characteristics without the specific problems of tellurium toxicity.

The range of heavy metal oxide (HMO) glass compositions has been enlarged by introduction of mixed anion systems, e.g. oxyhalides [1]. Most researches on oxyhalide glasses are based on heavy metal oxide formers that have lower liquidus temperatures, e.g. MoO_3, TeO_2, and Sb_2O_3 [2-4]. Multicomponent systems containing $PbCl_2$ appear to be most stable [5-7]. The vitreous network of the corresponding glasses is based on MO_4 tetrahedrons and also on MO_6 octahedrons (in trioxide-rich region). Large amounts of Sb_2O_3 in the glass-forming region indicate that vitreous network is formed by antimony coordination polyhedrons. These polyhedrons are not simple tetrahedral; they are less symmetric as they contain a lone pair electrons which need a volume close to that of O_2- ions [5]. The antimony oxide based glasses may have similar physical characteristics and applications as tellurite based glasses without problems related to tellurium toxicity [8].

Although Sb_2O_3 is glass-forming oxide it does not form pure glass. Admixture of typical modifiers (e.g. Li_2O) stabilizes the glass. In terms of structure the Sb_2O_3 creates mainly structural unit [SbO_3] in mentioned glasses. It is the tetrahedron consisting of three atoms of oxygen with antimony atoms diverted from equatorial plane. In General, the interruption of the connection between structural units arises when oxide of modifier (Li_2O, Na_2O, K_2O) is going to glass network. But in the case of these glasses, their stiffness is not significant when you add modifiers. This is evidenced from the fact that atoms of alkali metals support the creation of more co-ordinated structural units [SbO_6] or [SbO_4] [9]. Therefore, the glasses based on Sb_2O_3 are stable in spite of the high content of alkali metal oxide (fig.1) [10].

In contrast to conventional modifiers based on the alkali metals oxides and alkali earth, the PbO can form stable glasses in a wide concentration range (fig. 2) because of their possible dual role. In the first as the modifier [11] if the Pb-O binding is ionic and in the second as net-forming if Pb-O binding is covalent. Pb ions create three-dimensional spatial network if they are present in the glass as net-forming. This fact makes it possible to form a glass up to 90 mol % of the PbO content [14].

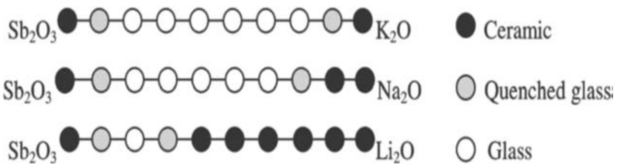


Fig. 1. Binary glasses in the Sb_2O_3–M_2O system [10].

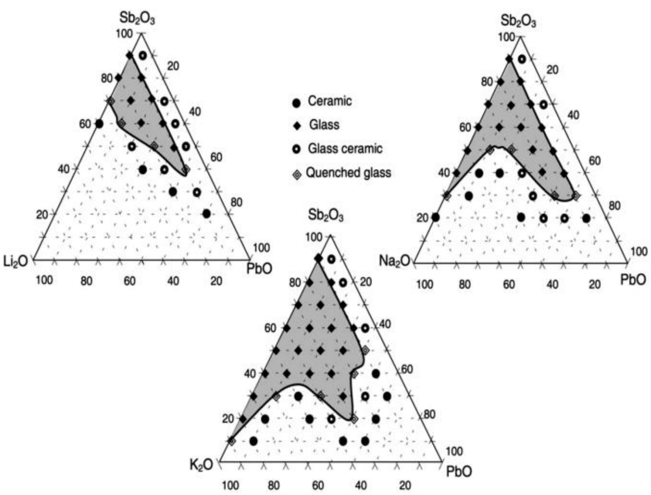


Fig. 2. Glass formation in ternary systems Sb_2O_3–M_2O–PbO. M = Li, Na, K [10].

This behaviour of Pb is caused by the electron structure of the Pb^{2+} ion. In fact, easily polarizable electron structure of Pb^{2+} ions strongly interacts with a very polarizable O^{2-} ion and it leads to the formation of covalent binding of Pb-O [15-17]. In this case, the PbO may create a structural unit [PbO_4] in the glass with the shape of tetrahedron and lead atoms in the middle [14].

In ternary glasses (90-x)Sb_2O_3 - 10Na_2O - xPbO substitution of Sb ions for Pb ions reduces the elastic properties of the glasses, which correlates with the decreasing of glass transition temperature values T_g. Pb ions occupy the places of Sb cations and create weak bindings Sb-O-Pb. Mentioned bindings are more deformable, which excludes occurrence of less deformable structural units [PbO4], i.e. there is the assumption that the PbO operates in glasses Sb_2O_3 - M_2O - PbO as modifier [18]. $PbCl_2$ operates also as modifier in the glass systems Sb_2O – $PbCl_2$ [6, 7, 19, 20].

It has been discovered that optical transmittance measured in the wave range of (3.75 – 8.00) μm depends significantly on the amount and the nature of defects of the prepared glasses and the glass composition. The highest values of optical transmittance and direct conductivity, as well, were found for the glass of the composition of 70Sb_2O_3 – 30$PbCl_2$ [21].

Because of high sensitivity of optical transmittance to glass composition we present sophisticated processing of experimental data to obtain a way for direct prediction of the proper glass composition-transmittance relation.

2. Artificial neural networks computation model

Application of methods for mathematical simulations and interpretation of optical, electrical, dielectric, mechanical and thermo-physical properties was at the forefront of material research in recent years [22, 23]. Artificial neural networks use the distributed parallel processing of information during calculations, it means that information recording, processing and transferring are carried out by means of the whole neural network, and then by means of particular memory places. The basis of mathematical model for the neural network is a formal neuron which describes by a simplified way a function of a biological neuron by means of mathematic relations (Fig. 3).

A neuron consists of a body, called a soma, which contains the input transmission channel in the form of dendrites; the output is provided by axon [24]. The formal neuron has n generally real inputs $x_1,\dots,x_n$ corresponding to dendrites. All inputs are assessed by appropriate synaptic weights $w_1,\dots,w_n$ which are generally also real. Weights determine the transmission rate of the input signal. The weighed sum of input values presents the inner potential of the neuron z [25]:

$$z = \sum_{i=1}^{n} w_i x_i - h \quad (1)$$

Output (state) of the neuron y modelling the electric impulse of the axon is generally given by a non-linear transfer function σ, the argument of which is the inner potential of z:

$$y = \sigma(z) \quad (2)$$

Learning is a basic and essential feature of neural networks. Knowledge is recorded especially through the strength of linkages between particular neurons. Linkages between neurons leading to a "correct answer" are strengthened and linkages leading to a "wrong answer" are weakened by means of the repeated exposure of examples describing the problem area. These examples create a so-called training set [26].

Neural networks are suitable to be used for their learning Back propagation algorithms for all types of predictions. This algorithm is convenient for multilayer feed forward network learning which is created minimally by three layers of neurons: input, output and at least one inner (hidden) layer. Between the two adjoining layers there is always a so-called total connection of neurons, thus each neuron of the lower layer is connected to all neurons of the higher layer.

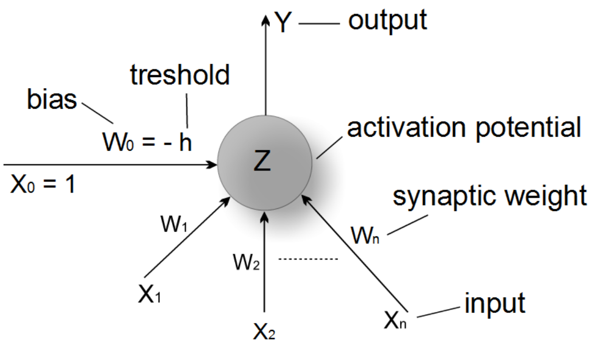


Fig. 3. Mathematical model of neuron.

Learning in the neural network is realized by setting values of synaptic weights between neurons, biases or inclines of activation functions of neurons. The adaptation at Back propagation types of networks is also called „supervised learning“, when the neural network learns by comparing the actual and the required output and by setting values of the synaptic weights so that the difference between the actual and the required output decreases [26].

Artificial neural networks are suitable for approximation of relations among various process data, among data with a high degree of nonlinearity and inaccurate data. This type of data often occurs in industrial processes of metallurgy. Neural networks are able to simulate behaviour of systems with very complex internal structure and complicated external behaviour, where analytic description is considerably complex; eventually it does not exist at all. They can simulate dependences which are solved with difficulties by classic methods of statistic data evaluation (e.g. regression analysis) and they are able to express more complex relations than these methods. Regression analysis requires at least partial knowledge of the internal structure of the system (system is known as a grey or white box), i.e. it is necessary to assess the structure of the regression function beforehand, which is difficult especially for multiparametric systems. Neural networks do not need knowledge of the system internal structure (system can be given as a black box); they are able to derive the dependency among variables just from the nature of the data by means of learning.

3. Experimental procedure

Investigated glasses were prepared from materials of high purity (> 98 %) at the University of Rennes in France. Production of glasses started by mixing of powders and their heating in quartz or quartz-glass ampoule went on while the transparent melt was gained. Then the melt was spilled at the brass plate [9]. Preparing glass by heating original carbonates ($PbCO_3$, Na_2CO_3, K_2CO_3, Li_2CO_3) one can notice their decomposition on CO_2 and the formation oxides, which create glasses [8, 10]. Temperature used during the glasses preparation depended on their composition and was from the interval (900 – 1100) °C.

Glass samples for electrical and dielectric measurements (cylinders of the diameter $\phi \sim 10$ mm and height ~ 1 mm) were coated by the conduct layers at the bottom. Temperature dependencies of direct electrical conductivity σ_{dc} at the heating rate 5 °C.min^{-1} were measured by picoammeter Keithley 6485 [27-29]

Measurements of temperature and frequency dependencies of complex electrical modulus M^* introduced by Macedo et al. [30] as a reciprocity of complex permittivity $M^* = (\varepsilon^*)^{-1} = M' + iM''$ were measured by GOODWILL LCR 819 device at the frequency range of (0.2 – 100) kHz [31]. Absorption spectra were measured by spectrometer CARLZEISS JENA JENATECH. The optical microscope JENATECH INSPECTION was used for the microscopic observation. The presence of the crystalline phase was found out by X-ray analysis by means of Philips PW 1710 device.

Temperature dependences of the dc conductivity σ_{dc} obey Arrhenius like relation

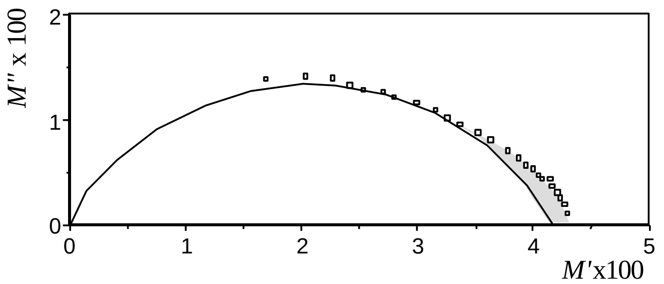
$$\sigma_{dc} = \sigma_o \cdot \exp(-U/kT), \quad (3)$$

where σ_o is a pre-exponential factor, U is a conduction activation energy, k is Boltzmann constant, and T is thermodynamic temperature.

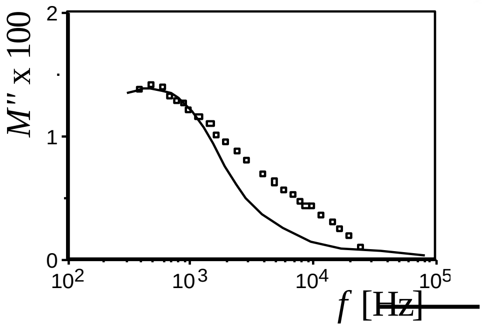
4. Results and discussion

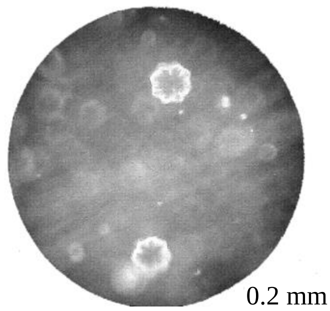
To find defects in samples we utilized the fact that they are non-transparent for the visible part of spectrum. The occurrence of defects was confirmed also by measurements of dielectric properties of samples [21] and measurements of their complex electric modulus M^* can be used (Figs. 4, 5), too. Presence of defects is apparent in the complex plane by the deviations from the ideal ordering on the periphery of the half circle. The amount of defects is detectable by means of the size of mentioned deviations as it can be seen from results obtained for samples SP 70 ($70Sb_2O_3 – 30PbCl_2$) and SKP 640 ($60Sb_2O_3 – 40K_2CO_3$ it is very non-homogeneous and thus opaque) - see the grey region in Figs. 4, 5. When the glass is opaque it contains substantial abundance of the crystalline phase (e.g. SKP 640) and two phases can be identify in its composition (Fig. 5). It can be realized by means of the superposition of data relating to the two single phases in the sample volume. X-ray measurements shows the abundance of the crystalline phase only for the sample SKP 640. The results confirm that the sensibility of the dielectric methods on the presence of the crystalline phase in glass is at least comparable to X-ray analysis (Fig. 6).

For the identification of crystalline phase and defects by means of measurements of a complex electrical modulus the change dielectric polarization of sample was monitored as a result of the occurrence of dipoles with a significantly shorter relaxation time, i.e. using higher frequencies (Figs. 4b, 5b).



a)





b) *c)*

Fig. 4. Dependencies measured for glass SP 70 ($70Sb_2O_3 – 30PbCl_2$) at temperature of 210 °C: a) Complex electric modulus (M'' vs. M') b) Dependency of the imaginary complex electric modulus M'' vs. frequency c) The microscope photo of glass made by polarized light

A high concentration of defects in mentioned glasses can be also detected by measuring of the dc electric conductivity [21]. In homogeneous glasses with a minimum percentage of defects the behaviour in accordance with the relationship (3) can be observed in the temperature dependence of dc electric conductivity, while the activation energy is U_1 = 1.1±0.1 eV. For example, this behaviour may be confirmed by means of results obtained for glasses SP 70 and 55 SP shown in fig. 7. In the case of glasses with high percentage of defects there is a deviation from the temperature dependencies described by the relations (3), or the activation energy decreases under U_1 = 0.7 eV. As an example of this behaviour, it can be mentioned results obtained for glasses SKP 640 and SP 40 shown in fig. 7.

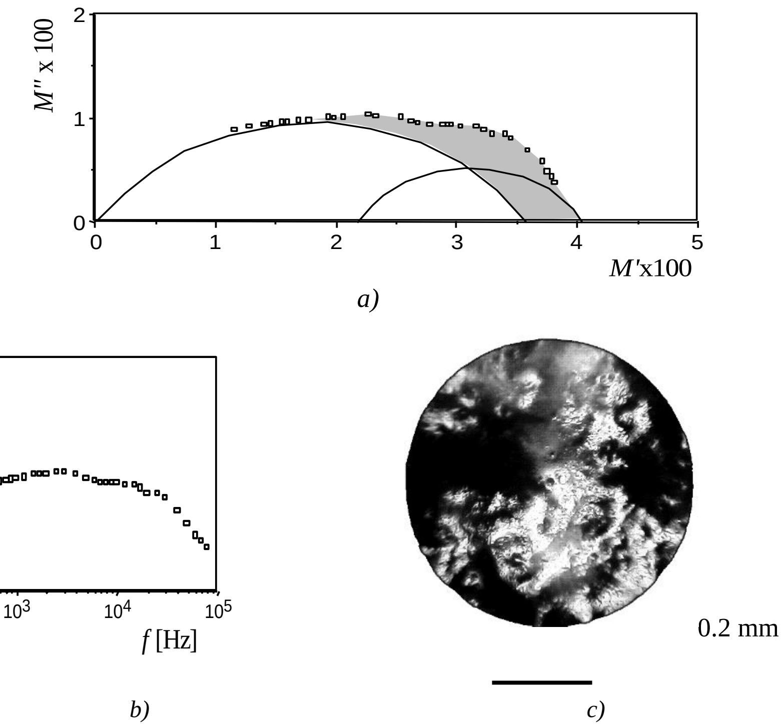


Fig.5. Results obtained for glass SKP 640 ($60Sb_2O_3 – 40K_2CO_3$) at temperature 210 °C: a) M'' vs. M' b) M'' vs. f c) The microscope photo of glass made in polarized light

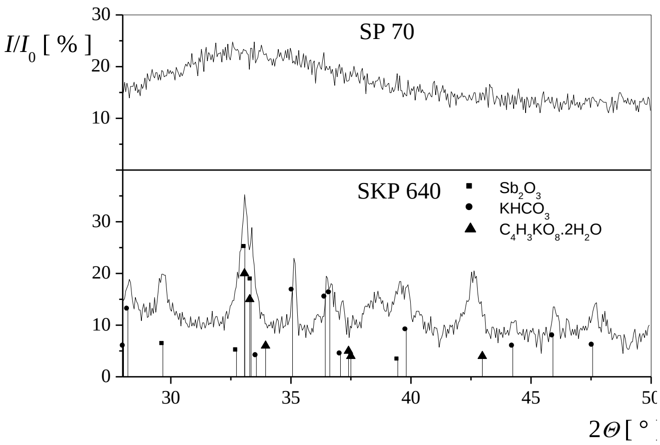


Fig. 6. X-ray analysis of glasses SP70 and SK 640 (λ = 1.7889 Å).

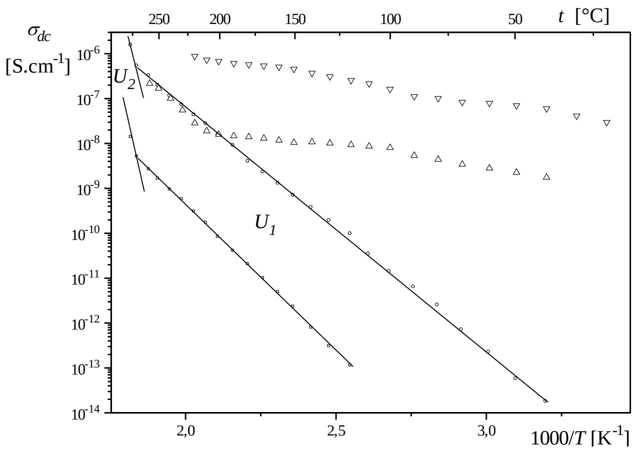


Fig. 7. Temperature dependencies of direct electrical conductivity σ_{dc} measured at temperature up to 280 °C: ○ SP 70 ($70Sb_2O_3 – 30PbCl_2$), □ SP 55 ($55Sb_2O_3 – 45PbCl_2$), △ SKP 640 ($60Sb_2O_3 – 40K2CO_3$), ▽ SP 40 ($40Sb_2O_3 – 60PbCl_2$)

Deviation from the behaviour described by the relationship (3) can be easily explained by means of the two-stages of the system at the presence of a significant percentage of defects with a comparable values of electric conductivity at room temperature per the glass phase and crystals, but with different values of the activation energy.

The reduction of activation energy and compliance with the relationship (3) can be explained by significantly higher value of dc conductivity characterizing a continuous network of crystals generated as a result of corrosion at the action of water. The removal of water from the volume (case SKP in fig.7) can change activation energy of the dc electric conductivity.

Optical properties determining the application possibilities of investigated glasses (IR area of the spectrum) are also significantly affected by the composition of glasses. System SP ($Sb_2O_3 – PbCl_2$) shown in fig.8 can be mentioned as an example. Optical properties are also influenced in a very small extent by the quality of the technology, i.e. by the occurrence of accompanying substances of crucible material (Si-O), and source substances (H_2O and CO_2) (fig. 9).

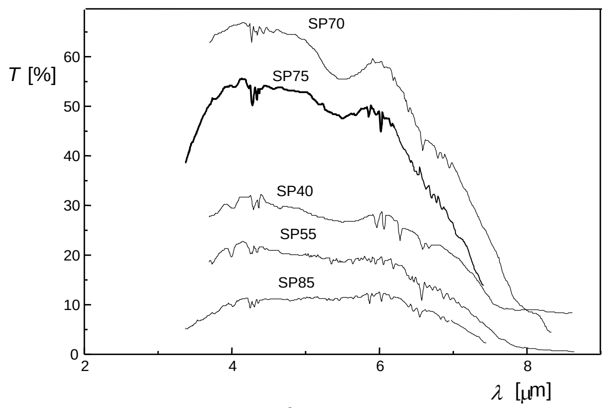


Fig. 8. Transmittance of glass system SP (Sb_2O_3 – $PbCl_2$): SP 70 ($70Sb_2O_3$ – $30PbCl_2$), SP 75 ($75Sb_2O_3$ – $25PbCl_2$), SP 40 ($40Sb_2O_3$ – $60PbCl_2$), SP 55 ($55Sb_2O_3$ – $45PbCl_2$), SP 85 ($85Sb_2O_3$ – $15PbCl_2$).

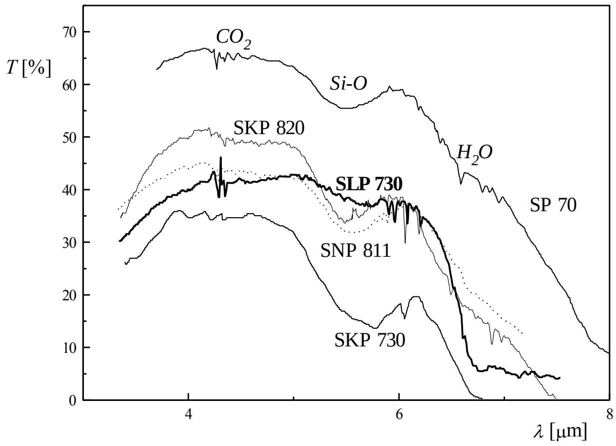


Fig. 9. Transmittance of glasses where absorption ranges CO_2, Si-O and H_2O are marked: SP 70 ($70Sb_2O_3$ – $30PbCl_2$), SKP 820 ($80Sb2O3$ – $20K_2CO_3$), SKP 730 ($70Sb_2O_3$ – $30K_2CO_3$), SLP 730 ($70Sb_2O_3$ – $30K_2CO_3$), SNP 811 ($80Sb_2O_3$ – $10Na_2CO_3$ – $10PbCO_3$).

A specific data file designed to create artificial neural networks included 13428 cases. Indeed 7 of the total number of variables were used as inputs to the artificial neural networks. As the input variables were identified concentrations of source substances Sb_2O_3, $PbCl_2$ $PbCO_3$, Na_2CO_3, K_2CO_3, Li_2CO_3 and wavelength. One output variable has been marked as the transmittance. Scheme of input and output variables related to the neural network is shown in Fig. 10.

Software Statistica – Neural Networks was used for the creation of artificial neural networks. Data file had to be modified before the creation of artificial neural networks so that it could be used in mentioned software (Statistica). Total amount of data were randomly divided into three parts: training, testing and validation. This is necessary for a proper learning and verification of the accuracy of the prediction of created artificial neural network. Several artificial neural networks with varying structure and parameters were created on the basis of adjusted data. The one that had the best results of learning has been selected for the prediction of defects. It was three-layer network with topology 7-10-1. This means that the input layer contains 7 neurons, hidden layer 10 and the output layer one neuron.

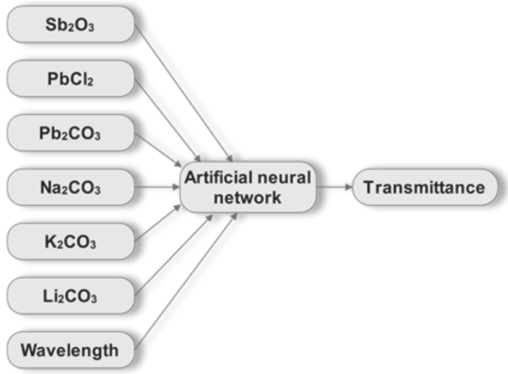


Fig. 10. Structure of input and output data.

The rate of inaccuracy between predicted and actual output represent a prediction error. In technical applications the error is mainly represented by following relations:

The relation for RMS error calculation (Root Mean Squared) – it does not compensate for used units

$$\mathrm{RMS} = \sqrt{\frac{\sum_{i=1}^{n}\left(y_i - o_i\right)^2}{n-1}} \tag{4}$$

The relation for REL_RMS error calculation – it compensates for used units

$$\mathrm{REL\_RMS} = \sqrt{\frac{\sum_{i=0}^{i=n-1}\left(y_i - o_i\right)^2}{\sum_{i=0}^{i=n-1}\left(y_i\right)^2}}\ , \tag{5}$$

where: n - number of patterns of a training or test set, y_i - predicted outputs, o_i - measured outputs.

Prediction errors of the chosen neural network model calculated according to relations (4) a (5) are RMS = 2.28 and REL_RMS = 0.068. Comparison of measured and predicted data is shown in fig. 11. Fig. 12 shows a histogram of the residues, expressing also the quality of the learning. Selected neural network enables to predict the optical transmittance with sufficiently small error.

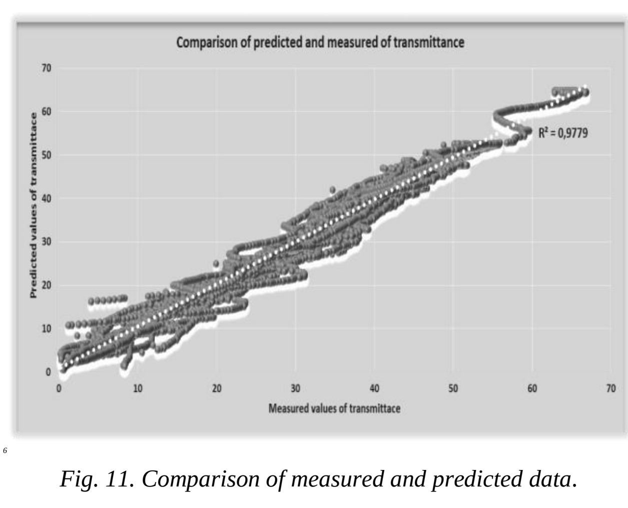


Fig. 11. Comparison of measured and predicted data.

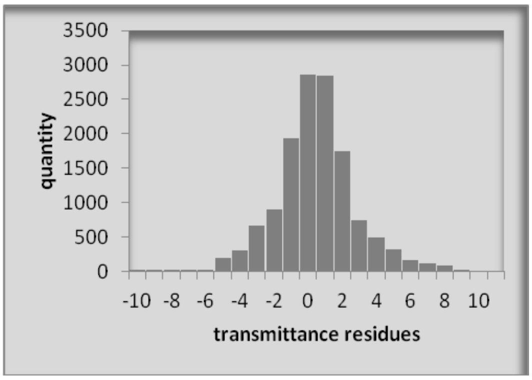


Fig. 12. Histogram of residues.

Furthermore, sensitivity analysis was realized for this neural network. This analysis reflects the influence of individual input variables on a given system. Sensitivity analysis showed that $PbCl_2$, Sb_2O_3 were variables with the greatest influence on the system. Conversely, wavelength and $PbCO_3$, Li_2CO_3, K_2CO_3 are variables, which have a relatively small effect on the system. The overview of sensitivity analysis and the significances is in the Table 1.

Table 1. Sensitive analysis.

Variable	Relative importance of the variables	Sequence
$PbCl_2$	119,62	1
Sb_2O_3	103,10	2
$PbCO_3$	70,10	3
Na_2CO_3	59,91	4
K_2CO_3	49,77	5
Wavelength	27,22	6
Li_2CO_3	25,11	7

For the use of the results of the learned artificial neural network parameters and the structure of the selected network were implemented to a custom program created in the development software environment for C++. This program is independent on the software Statistica, i.e. program in which the neural networks were created and tested. This program can be used for prediction of defects on the basis of the entering of input parameters to all required variables (Fig. 13).

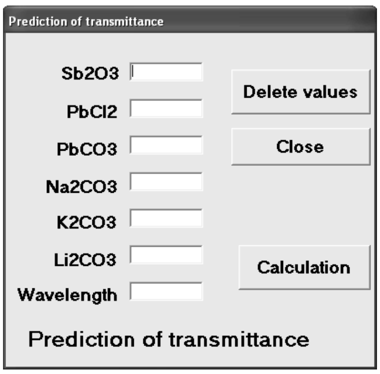


Fig. 13. Created application for prediction of transmittance.

In terms of conclusions about the plausibility of the ANN analysis, it is important to find out how the contents of individual components of used modifiers affect properties of glasses. Investigated glasses were prepared by melting method with the consequent abrupt cooling into metallic forms. In General, they may also contain a small amount of defects (bubbles, crystals) given by used laboratory procedure. The formation of bubbles is usually a remnant of decomposition process of starting substances based on carbonates, or decomposition of $PbCl_2$. The formation of small crystals is characteristic for the glass with a high content of Sb_2O_3. Pure Sb_2O_3 does not form a glass and if rough material (melt before refrigeration) is insufficiently homogeneous, it may arise locally crystals Sb_2O_3. Significant contamination of the system by the material of used crucible is the risk in case of the long run of homogenizing. Implementation of diagnostics of the impact of individual starting components summed up in table 1 shows that ANN is a convenient tool for further improvement of optical transmittance in the IR area of spectra. Sb_2O_3 is opposition to others starting substances ($PbCl_2$, K_2CO_3, Li_2CO_3, Na_2CO_3, $PbCO_3$), which modify the glass network. With regard to the role of Sb_2O_3 it is understandable its high importance (table 1).

The impact of other substances on optical properties and so their importance may be hardly verified without additional experiments aimed at identifying the type of structural units and location of modifying atoms. This task is very difficult, especially in case of a larger number of starting substances.

However, the realized ANN analysis (table 1) showed a new possibility for the determination of the relative importance which is in good agreement with the assumptions. High relative importance of starting substances containing Pb ($PbCl_2$, $PbCO_3$) is a given by the fact, that atom of Pb in the role of modifier interrupts structural units given by the Sb_2O_3. With regard to the coordination number 6 and the size of an atom of Pb in this arrangement it can be expected significant change of optical properties after the addition of the substances based on lead. The most significant relative importance at $PbCl_2$ is given by the fact that the chlorine atom may also participate in the interruption of bindings between structural units by the occupying positions of oxygen atoms in the structural units. The relatively small impact of the substances bringing alkali metals oxides (K_2CO_3, Li_2CO_3, Na_2CO_3) is associated with a small radius of the alkali metals. The relative lowest importance of Li_2CO_3 on the optical properties of the glasses is connected with radius of Li which atoms occupy in the glass network primary interstitial positions. If we start from table 1, we can say that the wavelength is also a low important factor, which is in accordance with the assumptions.

5. Conclusion

The measurements of the temperature dependencies of the direct electric conductivity show the strong influence of the concentration of the individual glass compounds of systems Sb_2O_3 – $PbCl_2$ and Sb_2O_3 – PbO – M_2O (M is Na, K, Li) on their electric and dielectric properties. Glasses show the same mechanism of the electric conductivity with activation energy, which goes to the value 3.75 eV when temperature is higher than 250 °C.

Similarly optical transmittance *T* of systems Sb_2O_3 – $PbCl_2$ and Sb_2O_3 – PbO – M_2O depends strongly on the glass composition and the amount of defects, too. The glass $70Sb_2O_3$ – $30PbCl_2$ reached the highest value of *T*. The minimal content of defects in its volume includes this glass among the perspective ones for next development.

The measurements of the complex modulus *M** of mentioned glasses showed their high sensitivity to the changes of glass structure connected with the creation, the sort and the amount of defects. The sensibility of the used methods is comparable with the usual exploited methods (X-ray analysis, optical microscopy) and makes possible to assess partially the quantitative occurrence of defects in the glass volume.

A model of neural network for prediction of the optical transmittance was created. Model enables to predict the transmittance with sufficiently small error. After evaluation of results we can state that exploitation of neural networks is advantageous, if it is necessary to express complex mutual relations among sensor-based data. Neural networks are able to realize and appropriately express general properties of data and relations among them and on the contrary to suppress relationships which occur sporadically or they are not sufficiently reliable and strong. Their usage enables retrieval of relationships among parameters of the process which with use of common methods are not possible to trace for reason of their mutual interactions, big amount and dynamics. Use of a neural network seems to be a suitable tool for estimating different important optical parameters.

Acknowledgement

This work was supported by the Slovak National Science Foundation under grants APVV No. SK-FR-2013-0007, APVV No. SK-CZ-2013-0182, VEGA No.1/0356/13 and VEGA No.1/0184/14.

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