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Rapid Particle Size Analysis of Suspensions Based on Video Technology and Artificial Neural Network with Additional Training During Operation

Vladimir S. Fetisov, Zarina I. Kharisova, Oleg A. Dmitriyev and Olga V. Melnichuk

Ufa State Aviation Technical University,12 K. Marx st., Ufa, 450008, Russia.

Abstract

The original system for rapid determination of suspensions particle size distribution is proposed. The system is based on video technology and artificial neural network (ANN) that has training procedure not only in time of initial calibration but during operation too. The system structure and functioning algorithm has been considered. Results of some experiments are shown. The proposed system has an on-line laser nephelometric analyzer based on a simple measurement chamber with transparent window. The image of scattered light is registered by the camera. Further treatment converts some special parameters of images into input variables of the ANN which continuously generates parameters of particle size distribution on its outputs. The ANN learning is implemented on the basis of the reliable (but not so fast) microscopic method which is switched in use only in some moments when the registered image is utterly different from the known ones. Such approach allows to perform rapid analysis and to maintainits high metrological reliability.

Keywords: Suspension, Particle Size Distribution, Granulometry, Nephelometry, Artificial Neural Network, Additional Training During Operation

INTRODUCTION

Using up-to-date technologies in the chemical, paint, food, pharmaceutical and other industries it is essential to determine particle size distribution of different suspensions. The particle size distribution is understood as percentage (share) distribution of particles' mass or particles' number according to their sizes (ranges of sizes) [1]. The tools for such analyses should work in mass production conditions and to be fast enough to ensure the usual process control. For example, timely information on the current particle size distribution of some powder or some suspension can be used to change the operating parameters of process equipment such as dispersers, mills, filters, centrifuges, etc. in order to ensure final product quality [2]. In the most of industrial processes today the determination of particle size distribution in the range of 0.1-100 microns (µm) is required.

The conductometric method, varieties of sieving and sedimentation methods [3] and the group of optical techniques: laser diffraction, nephelometry, turbidimetry, light microscopy can be understood as traditional methods for particle size analysis [4].

Conductometric method (method based on the Coulter counter) is thought to determine the electric resistance while the investigated particle-hole (capillary) is passing by. The voltage pulse is measured, which amplitude is proportional to the volume passing through the particles capillary. Then the obtained data is processed, the number of particles is counted. Such modern tools allow to analyze the particle sizes in the range from µm to millimeter. This method can be applied only to suspensions, it is very sensitive to particle concentration and requires prior preparation of the studied suspension.

Sieving method is quite simple to implement and is used mainly for dry materials with particle sizes from 5 µm and larger. Sedimentation methods are used to determine the dependence of the particles deposition rate on their size, recognizing gravitational sedimentation (under gravity) and centrifugal (under the influence of centrifugal forces). The dispersed systems analysis based on centrifugal sedimentation reduces significantly the time of its implementation compared to the gravity method, furthermore, it allows to study particles of submicron size. This method has a low implementation cost, however, the sedimentometric measurement process takes a long time, being their main disadvantage.

The laser diffraction method is based on the principle of angular light scattering when a laser beam passes through the dispersed system [5]. This method allows to determine the particle sizes in the range from 10^{-9} to 10^{-2} m. Larger particles scatter light mostly from low angles, relatively small particles do this at large angles. The method is complex to implement, in addition it is necessary to know the optical properties of the samples to precisely examine the system dispersion [6].

Nephelometric and turbidimetric methods determine the particle size by the intensity of dispersed system light scattering. Optical analyzers based on these methods can be constructed either as a turbidimeter where the photodetector is located on the radiation axis and it detects the transferred attenuated radiation, or nephelometer where the receiver is located at an angle to the radiation axis and it detects scattered radiation [7]. These methods are often used to construct on-line measuring instruments, as their main advantage is almost instant results.

The easiest and most applicable for liquid disperse media way of sizing the particles is microscopic analysis based on the direct counting method with the ability to determine the shape of the studied particles. The operating principle of devices based on microscopy includes using optical means to photograph the studied samples, with the subsequent images processing [8, 9]. Microscopic methods are traditionally divided into the submicroscopic (particles in the range from 1 nm to 1 μm) and light methods (for particles in the range from 1 μm to 1 mm), however, up-to-date digital microscopes with high-resolution cover both ranges and determine the size of particles of the traditional microscopic methods. Providing high reliability of the results the microscopic methods are often carried out in laboratories and are difficult to integrate into technological processes.

Currently, neural network technology are often used besides the technical means for particle size distribution determination and the microscopic methods [10], allowing to process images of samples by a special neural algorithms in order to classify particles size and shape. Neural networks can replace complex math by simple "input-output" mapping that is tuned in the process of preliminary network training by the examples.

Presentation of the results using all these methods ultimately implies the construction of histograms of the particle size distributions, as well as integral and differential curves which graphically describe the particle size distribution and characterize the relative content of particles of a size (density distribution), respectively. It is quite easy to obtain the results of particle size analysis in the laboratory, however, the study of suspension dispersion in conditions of the process flow requires special solutions to provide both sufficient measurement reliability and result timeliness.

The purpose of the present work and the related research is to provide rapid determination and acceptable quality of particle size distribution of suspensions in flow conditions on base of video technology and artificial neural network with additional training procedures during its operation. The proposed technical solutions allow to combine the advantages of the nephelometric and microscopic methods as well as to achieve acceptable performance and automatic continuous maintenance of the metrological reliability.

THE STRUCTURE OF THE PROPOSED SYSTEM AND ITS FUNCTIONING ALGORITHM

The rapid analysis of particle size distribution by direct environment photographing in the flow conditions is very difficult to carry out. The existing systems can be successfully applied for the analysis of coarse particulate materials, however, they are practically impossible to use for the particle size distribution analysis of the liquid dispersed media because in this case the camera should operate in the mode of flow microscopic photography, which makes measurement extremely unreliable (due to contamination of the camera optics, or possible defocus, etc.). In such conditions a variety of photometric analyzers can work, namely nephelometric analyzers based on photodetector matrices [11] potographing the scattered light pattern in the studied environment which then can be converted into information about the particle size distribution basing on the theoretical models.

Figure 1 shows the structure of the proposed system. The diagram shows: 1 – process chamber with a controlled suspension; 2 – concentration normalizer; 3 – flow-through measuring chamber with its input 4 and output 5 nozzles; 6 – laser emitter with a rigid light guide 7; 8 – light trap; 9 – drain system; 10 – camera; 11 – pattern selection module; 12 – neural network module; 13 – results indicator; 14 – objective analysis module (microscopic analyzer); 15 – control module; 16 – controlling valve.

The flow-through measuring chamber 3 is the most important part of the system. It is located in such a way that the inlet 4 is directed downwards, and output 5 one is directed up and is connected with a drain system 9. Thus, the controlled suspension continuously flows from the bottom up, which prevents particles settling. To eliminate the stagnant zones, it is recommended to round the corners of the square section of the chamber. The flat front panel of the chamber 3 is made transparent and turned to the camera 10, and laser light through a thin optical fiber 7 is transferred into its center. This method of input radiation is necessary to registrate radiation scattered in all directions including the back direction. The light scattering pattern recorded by the video camera 10 depends on particle size distribution of the suspension.

The experimental model of the flow measurement chamber is shown in Figure 2 with the same numbers of elements as in Figure 1.

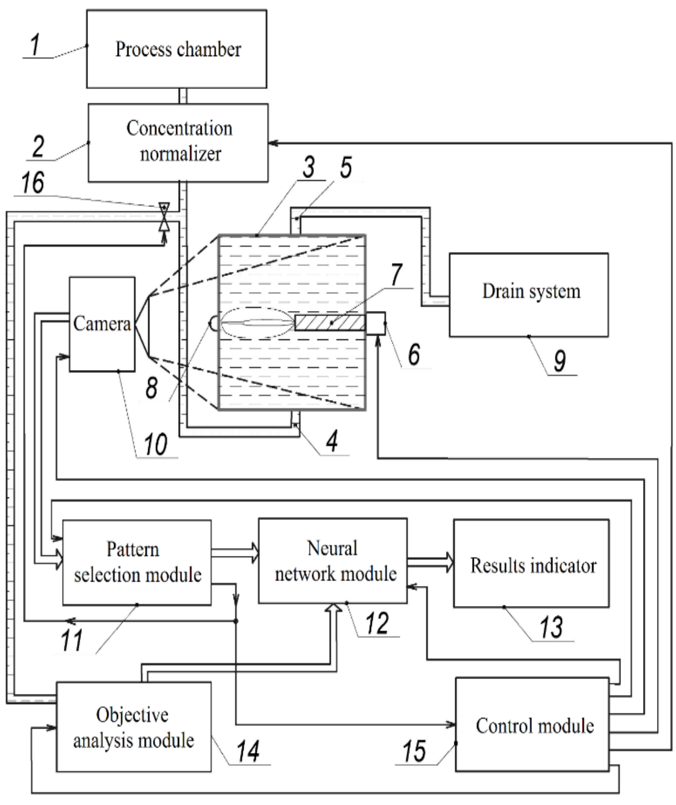


Figure 1. The structure of the proposed system

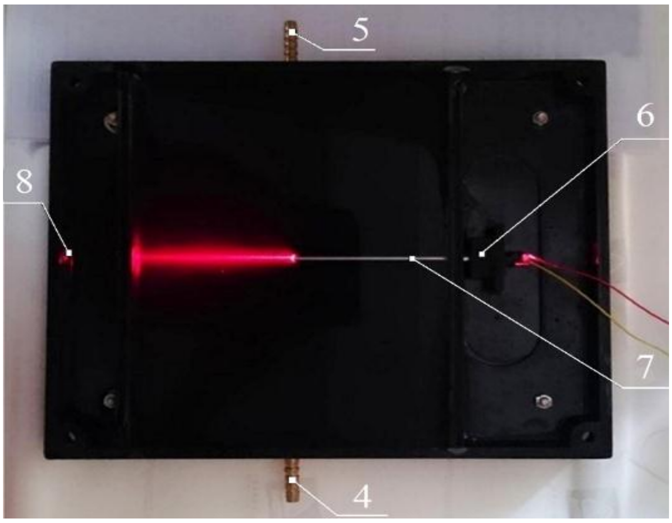


Figure 2. The experimental model of the flow measurement chamber

The optimal size of the measuring chamber 0.1x0.1x0.01 m was identified using a specially developed mathematical model of scattering radiation in the measuring chamber for the given emitter parameters (the wavelength being 680 nm, the emitter window area being 10^{-6} m^2, the output intensity being 1000 W/m^2) and environment (the number density being 108..109 particles/m^3 and the average particle size being 1 µm).

In the preliminary mode (basic calibration) some liquid samples with a certain and highly different particle size distribution are sequentially transferred from the process tank 1. After the special signal from the control module 15 each sample passes through the concentration normalizer 2 which produces a dilution of the incoming sample and brings its concentration up to a certain standard values. Then the suspension enters the chamber 3 and flows further to the drain system 9. By the signal from the control module 15 the laser 6 emits a beam which shines through the studied sample and dissipates in different directions. The direct incident beam falls into a light trap 8 which prevents the reflection of the beam back into the chamber from its walls. The light scattering pattern is recorded by the video camera 10 and shot by shot in the form of arrays of pixel brightness values is sent to the pattern selection module 11 which analyses a set of the most general features of the image. If the pattern selection module 11 detects a set of features that differs significantly from the already known and stored in memory one, then the valve 16 is opened, which provides the flow of the analyzed fluid to the objective analysis module 14 (the microscopic analysis is implemented in this block automatically, semi-automatically or in manual mode). The analysis having been finished, the numerical results characterizing the particle size distribution, are transferred to the neural network module 12 for its training. The results are transferred to the results indicator 13 where a diagram of particle size distribution is constructed. After network training for all the basic samples the cycle of the initial training is ended.

In the main operation mode (measurement mode) all the modules described above work similarly with the only difference that the objective analysis module 14 is in the standby mode most of the time, and the neural network module 12 produces a virtually instantaneous transformation of the signals array from the pattern selection module 11 into the results of particle size analysis by the previously trained artificial neural network. And only in certain moments of time when the pattern selection module 11 detects the new feature set coming with the input image, it generates a control signal initiating a new objective analysis, and then the additional training is carried out on new samples, that contributes to the gradual increase of the accuracy and metrological reliability of granulometric analysis.

THE RESULTS OF THE EXPERIMENTS

Some aqueous suspensions of synthetic diamond powders of different fractions (from 0.5 to 40 µm) with rounded particles were studied during the experiments. The samples with the given concentration (1 gram/liter) and particle size were investigated sequentially in the flow measuring chamber. A scattering pattern photographed by the camera was recorded for each sample and microscopic images were made (Figure 3). The microscopic analysis was performed using a digital microscope Trinocular MZJG160C by Amscope company and the related software. The obtained images were processed in the NI Vision Assistant program of Vision Development Module of LabView 2010 software package (National Instruments) designed to process and analyse images and to generate automatic codes for LabVIEW. The number and sizes of particles were counted using the images. The data obtained were used to construct histograms of the particle size distribution as well as differential particles distribution curves for each of the samples (column 4 on Figure 3). Besides, the analytical functions of density of particle distributions were also obtained. Particle size distribution corresponds to the gamma distribution curve. The analytical function of the gamma distribution in differential form is given by [12]:

$$\rho(D) = \begin{cases} \dfrac{\lambda^{\alpha} \cdot D^{\alpha-1}}{\Gamma(\alpha)} \cdot e^{-\lambda \cdot D}, & if\ D \geq 0 \\ 0, & if\ D < 0 \end{cases}$$

where D is particle size, $\Gamma(\alpha)$– gamma function, parameters $\alpha > 0$, $\lambda > 0$.

In practice, the form of the particle size distribution is easier to estimate using geometrical parameters of the differential distribution curve (probability density function) which can be used as the output variables for the ANN training (it can be limited to three parameters: the mode M_0 ($M_0=(a-1)\cdot\lambda$, $a \geq 0$), the dispersion D_0 ($D_0=a\cdot\lambda^2$) and the skewness K_a ($K_a=2/a^{1/2}$)).

The results of the experiments for 8 samples were put into an observation table (Table1) where for each range of particle sizes corresponds to the results of the microscopic analysis (the three values of M_0, D_0, K_a). The image of the scattered light was shot 10 times for each sample. The data was then used to train the ANN.

International Journal of Applied Engineering Research ISSN 0973-4562 Volume 12, Number 7 (2017) pp. 1271-1278

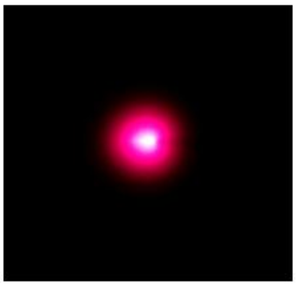
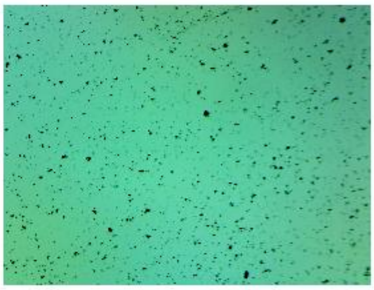
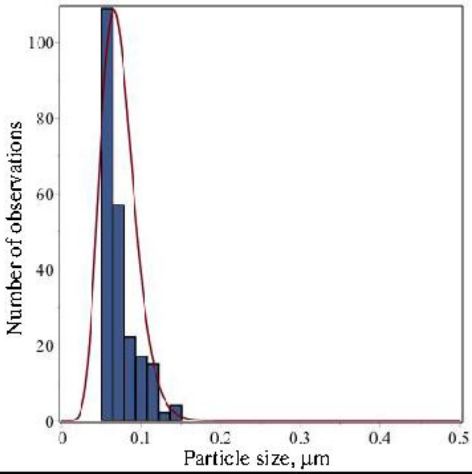
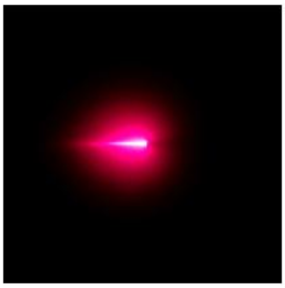
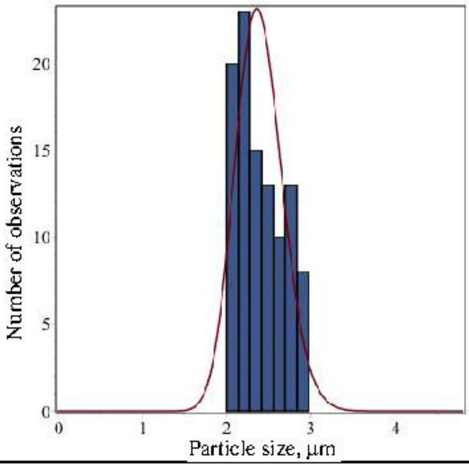
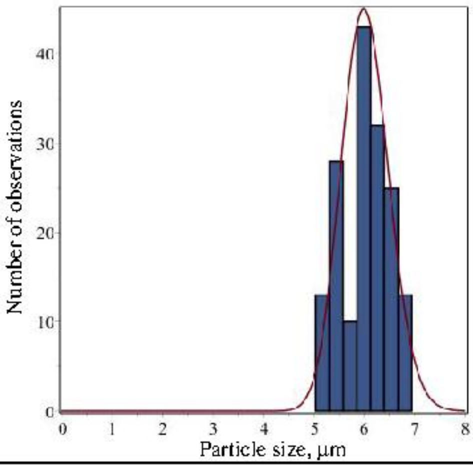
Particle size range	Pattern of radiation scattering	Microscopic picture	Particle size distribution
0 - 0.5 µm			
2 - 3 µm			
5 - 7 µm			

Figure 3. Patterns of radiation scattering and the corresponding particle size distributions

Table 1. Results of microscopic analysis transformed to parameters M_0, D_0, K_a

Number of sample	Particle size range, µm	The Average Values of the Distribution Parameters		
		M_0	D_0	K_a
1	0 – 0.5	0.072064	0.000464	0.59768
2	0.5 –1	0.70175	0.020168	0.40469
3	2 – 3	0.076625	0.076625	0.23149
4	5 – 7	6.0257	0.22151	0.15614
5	14 – 28	24.742	7.004	0.21391
6	28 – 40	31.471	7.638	0.17565
7	Composition 0.5 – 1; 2 – 3	0.97753	0.35223	1.2142
8	Composition 14 – 28; 28 – 40	23.514	8.6839	0.2508

TRAINING OF THE ARTIFICIAL NEURAL NETWORK

The well-known neural network of MLP type (multilayer perceptron) was used to process the captured images. The training algorithm was Back Propagation [13].

Being obtained in experimental studies, the radiation scattering patterns were processed in the LabView program which allows to determine the pixel brightness values at specific points and save them in a special file. The special points of the image were determined as follows. The field of scattering was evenly divided into a few concentric zones. On the borders of these zones the points were automatically made with an interval of 10 degrees (Figure 4). The obtained 180 brightness values at specific points were the input variables for the ANN.

The mentioned distribution parameters, namely the mode M_0, the dispersion D_0 and the skewness K_a were the output variables for the neural network.

Neural network training was performed in software package STATISTICA Neural Networks 4.0. The MLP structure containing 180 neurons in the input layer (according to number of input variables), 25 neurons in the intermediate one and 3 neurons in the output one (the number of output variables) was used (Figure 5).

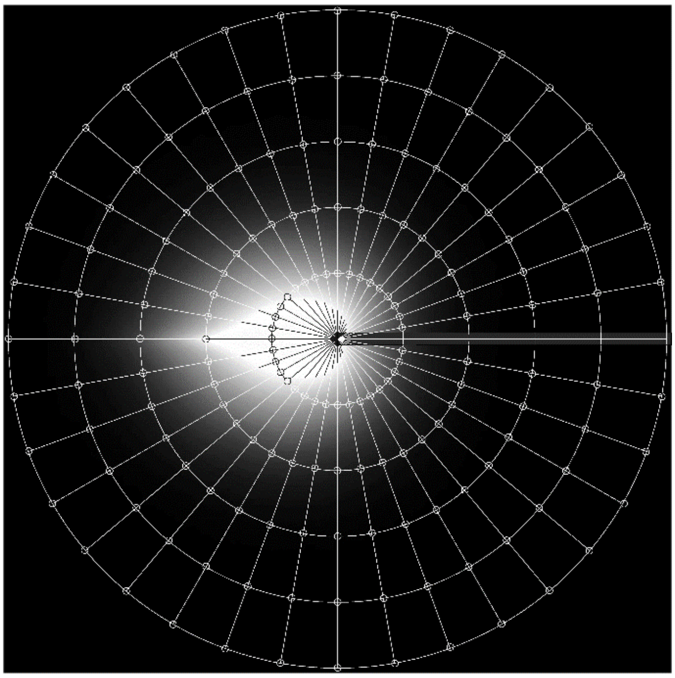


Figure 4. The special points for pixels brightness determination in the radiation scattering area

International Journal of Applied Engineering Research ISSN 0973-4562 Volume 12, Number 7 (2017) pp. 1271-1278

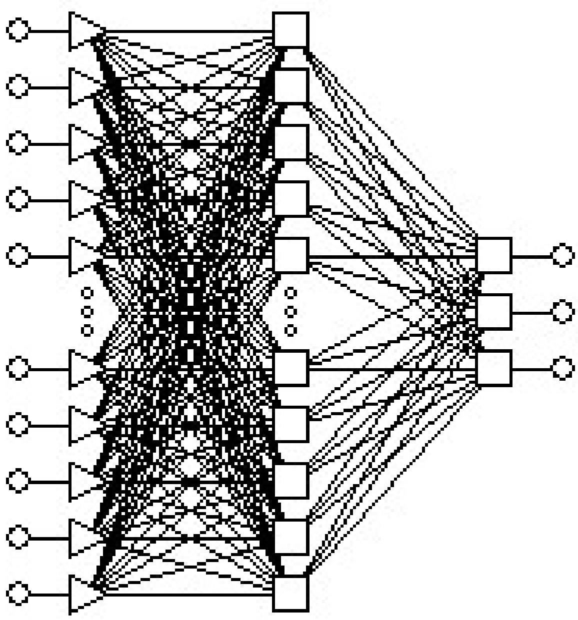


Figure 5. The structure of neural network: MLP with the number of neurons in layers 180-25-3

The observations data for 8 samples presented in Table 1 was used for training. As 10 shots of scattering pattern were made for each sample, one sample is presented with 10 observations. In total, the training table contains 80 observations (rows). A fragment of the training table is shown in Figure 6. The columns of the input variables VAR1-VAR180 correspond to the brightness of certain points of the scattering patterns and the columns of the output ones VAR181-VAR183 correspond to distribution parameters M_0, D_0 and K_a.

	VAR1	VAR2	VAR3	...	VAR178	VAR179	VAR180	VAR181	VAR182	VAR183
01	60	71	77		0	0	0	0.072064	0.000464	0.59768
02	56	63	68		0	0	0	0.072064	0.000464	0.59768
03	73	79	85		0	1	0	0.072064	0.000464	0.59768
04	60	66	70		0	0	0	0.072064	0.000464	0.59768
05	52	58	67		0	0	0	0.072064	0.000464	0.59768
06	58	70	78		4	7	8	0.072064	0.000464	0.59768
07	57	71	82		0	0	0	0.072064	0.000464	0.59768
08	77	89	103		0	0	0	0.072064	0.000464	0.59768
09	98	115	126		0	0	0	0.072064	0.000464	0.59768
10	99	110	121		0	0	0	0.072064	0.000464	0.59768
11	29	33	42		0	0	0	0.70175	0.020168	0.40469
12	27	32	37		0	0	0	0.70175	0.020168	0.40469

Figure 6. The dataset fragment for the neural network training
(VAR1-VAR180 are input variables, VAR181-VAR183 are output variables)

In each group of 10 observations for the used samples the first 6 observations were training ones, and 4 observations were verifying ones. Thus, the training selection was represented by 60% of all observations, and verifying selection was represented by 40% of all observations.

The training error (estimated by the verifying selection) is stabilized at a satisfactory level when the number of training iterations (epochs) is of about 1.000 (Figure 7).

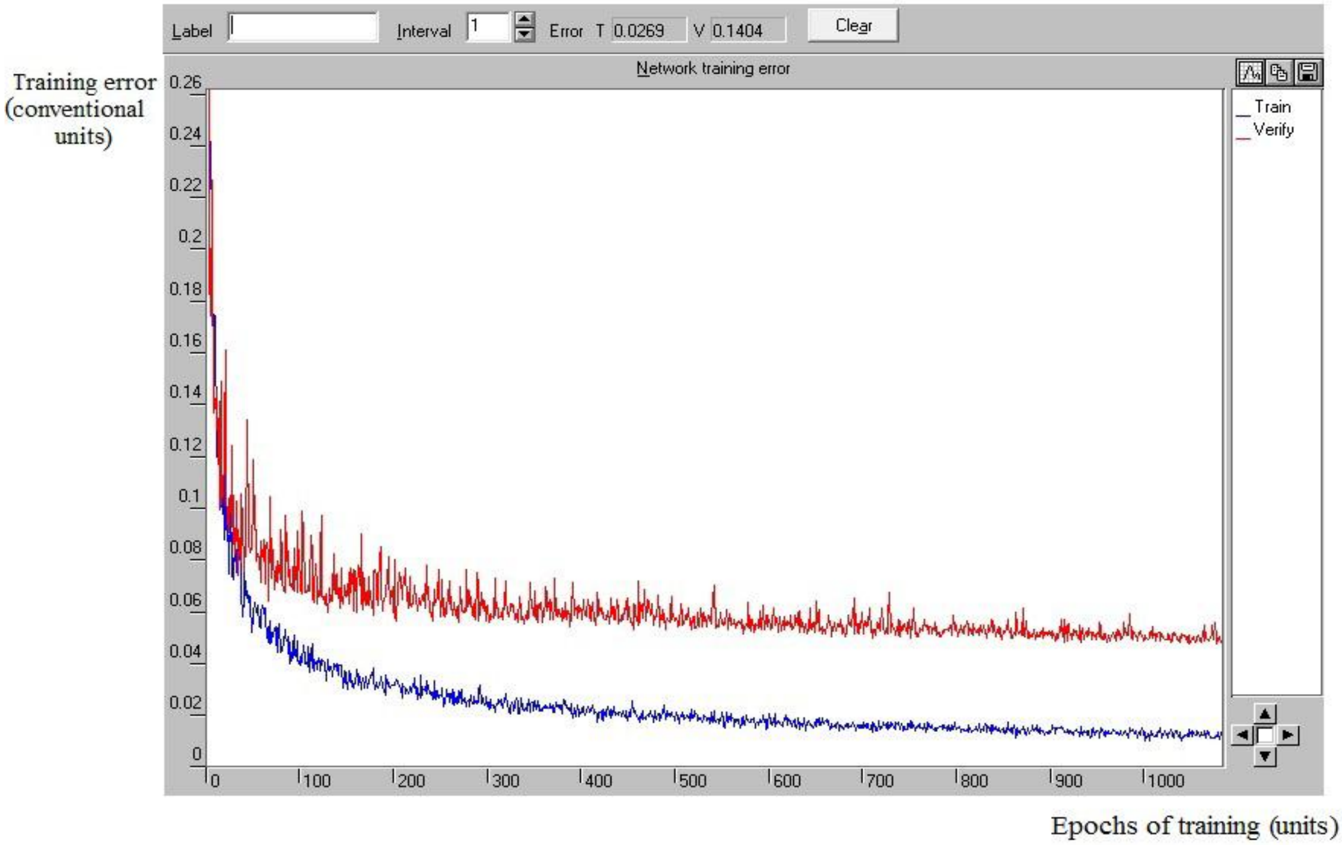


Figure 7. The training error on the training selection (lower graph) and verifying selection (upper graph)

CURRENT MEASURINGS AND TRAINING NEURAL NETWORK DURING OPERATION

In the mode of current measuring, if the continuously produced resulting scattering pattern doesn't differ much from the already known and stored in the memory of the pattern selection module, the ANN is simply used as the image processor, quickly converting the vector of brightness of pixels in 180 certain points into the vector of 3 particle size distribution parameters.

The process of comparing current vectors of input variables of the ANN with the same ones stored in memory of the pattern selection module and the further decision-making process are as follows: the vector of input variables (the mentioned 180 values of pixel brightness) are alternately compared with the same vectors for all the known samples stored in the memory. The comparison result is the similarity score computed as the sum of squares of differences between the pixel brightness values for the corresponding certain points of two images. The minimum value of this score demonstrates the greatest similarity of two compared vectors.

- The similarity score is compared with the threshold value, the choice of which depends on the required accuracy and acceptable frequency of additional trainings (the threshold value of 1000 reference units corresponds to high accuracy and frequently performed trainig procedures, the value of 10000 reference units corresponds to the average accuracy and frequency of the performed trainig procedures, the value of 100000 reference units corresponds to low accuracy and rarely performed trainig procedures). In case of exceeding the threshold of similarity score the pattern selection module produces the special signal indicating that the unknown sample is found and additional trainig procedure is required for the system. Simultaneously the vector of the obtained 180 variables is saved into the memory of the neural network.
- The objective analysis module (microscopic analyzer, operating in automatic or interactive mode) is started, analysing the obtained sample in detail and the values of M_0, D_0 and K_a are defined for the new training cycle of the ANN. They are stored in the memory of the neural network module as output variables.
- For better training on the obtained observation nine extra similar observations are created with artificial noise. But the output variables M_0, D_0 and K_a remain the same. Moreover, the first six observations are marked as training ones and the other 4 as verifying ones.
- The ANN training is conducted for all observations, the existing and new ones. There would be 90 observations in total for our case.

Below we consider the example demonstrating the effectiveness of the neural network training during operation.

The input of the neural network received the input vector corresponding to a new sample with previously unseen particle size distribution. This suspension was subjected to microscopic analysis and the following distribution parameters was determined: M_0=40.50, D_0=20.21, K_a =34.34. If to use the previously trained network without any additional training, it produces the result with a rather large error: M_0 =35.86, D_0=27.98, K_a=20.48. After the additional training the sample was re-examined, and the neural network gave a much more similar results (M_0 = 40.88, D_0 = 20.09, K_a = 34.24).

CONCLUSION

The proposed system possessing the performance of on-line nephelometric analyzer based on a simple measurement chamber at the same time has high metrological reliability supported through objective (microscopic) analysis tools switched in use if necessary.

The artificial neural network trained on the basic samples converts the obtained scattering patterns into the results of the granulometric analysis presented as three particle size distribution parameters: the mode M_0, the dispersion D_0, and the skewness K_a. The ANN training implemented during system operation contributes to the gradual improvement of the accuracy characteristics of the system and maintaining the reliability of granulometric analysis.

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