ANALIZE STATISTICE ŞI MODELE PREDICTIVE PENTRU CERAMICI COMPOZITE DENSE DE TIP Y-ZrO_2 – Al_2O_3

STATISTICAL ANALYSIS AND PREDICTION MODELS FOR YTTRIA-STABILIZED ZrO_2-Al_2O_3 DENSE COMPOSITE CERAMICS

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The most common objective of the statistical analysis is to explain the influence of random variations and events. Perhaps the most important objective is to manage simultaneous and combined effects of several influencing factors, when those factors can not be easily controlled in the experimental frame.

Multivariate Analysis – MVA is based on the principle of multidimensional statistics, which involves observation and analysis of several statistical variables at a time. This analysis is used to study several dimensions simultaneously, taking into account the effects of all variables on the characteristics of interest [1-4].

By introducing ceramic properties of some dense composite ceramics, zirconia – alumina type, with oxide additives for densification as independent variables, it was obtained a quality model. The model proved that it is necessary a more rigorous control of these properties in order to improve the compressive strength [5-6].

The developed statistical models are consistent with existing assertions in this area - they are viable, but also offers new insights - of interest from the point of view of optimizing the composition and properties of specific ceramics.

Cel mai cunoscut obiectiv al analizei statistice este acela de a explica influența variațiilor aleatorii. Probabil cel mai important obiectiv este acela de a descurca efectele simultane şi combinate ale mai multor factori de influență, în situații în care acei factori nu pot fi uşor controlați în cadrul experimentului.

Analiza multidimensională (MVA - Multivariate Analysis) se bazează pe principiul statisticii multidimensionale, care implică observarea şi analizarea mai multor variabile statistice într-un moment. Această analiză este folosită pentru a studia mai multe dimensiuni concomitent, ținând cont de efectele tuturor variabilelor asupra caracteristicilor de interes.

Prin introducerea proprietaților ceramice ale unor ceramici compozite dense de tip zirconă – alumină, cu aditivi oxidici pentru densificare, ca variabile independente obținem un model calitativ superior. Acest model dovedeşte că este necesar un control mai riguros al acestor proprietăți pentru a îmbunătăți rezistența mecanică la compresiune.

Modelele statistice create sunt în concordanță cu aserțiunile deja existente în acest domeniu - sunt viabile, dar oferă şi perspective noi - prezintă interes, din punctul de vedere al optimizării atât a compozițiilor cât şi a proprietăților specifice ceramicilor.

Keywords: *statistics, ZTA properties, prediction, dopants*

1. Multivariate analysis

Statistical analysis is seen by experts as a necessary evil to public scientific articles, as another way to influence friends and enemies to a certain perspective, but there is a group saying that it is an irrelevant deviation from the real science. Statistical tools serve these purposes only to few people. However, in the right hands, they can be very useful to scientific purposes, which is an important step toward understanding the mechanisms underlying those scientific data. Another positive aspect of the statistical analysis is to allow optimization of processes and compositions specific for certain materials or systems.

The most common objective of the statistical analysis is to explain the influence of random variations.

Perhaps the most important objective is that to manage simultaneous and combined effects of several influence factors in the situations in which those factors can not be easily controlled within the experiment [1 - 7].

Multivariate Analysis – MVA is based on the principle of multidimensional statistics, which involves observation and analysis of several statistical variables at a time. This analysis is used to study several dimensions simultaneously, taking into account the effects of all variables on the characteristics of interest [8].

1.1. Prediction

In the scientific context, a prediction is a rigorous estimate, usually quantitative, anticipating what will happen under certain conditions. This is done through repeatable experiments or observational studies.

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A scientific theory whose assertions are not consistent with existing observations and evidence will be denied. Predictions remain untested theories containing proto-science until their test methods become known to community. In addition, when new theories generate many predictions, they are very popular because they can be confirmed or refuted quickly and easily. In most scientific fields, desirable theories are those that predict a large number of events from few basic principles.

1.2. Methods

In statistics, correlation (often measured as a correlation coefficient) indicates the direction and strength of linear relationship between two random variables. The capability of multidimensional analysis procedure determines the probability that elements characterized by two or more variables falls in special technical conditions certified. When variables are correlated is important to consider their overall behavior, because treating each variable separately one can obtain deceptive information on overall system capability.

Method of least squares builds a statistical model linking X independent variable with Y dependent variable. The procedure is very useful when there are several factors and the main objective is the prediction of the dependent variables (response variables). Multiple regression procedure determines a statistical model that describes the impact of two or more quantitative factors X on a dependent variable Y. The model can be used to make predictions, but also to determine the limits of certainty. Residual elements (outliers) can be identified and also discovered the values / data sets with a major influence [8-10].

Canonical correlations help to identify associations between two sets of variables by finding linear combinations of the two sets of variables showing strong correlations. Pair of linear combinations with the strongest correlations forms the first set of canonical variables. The second set is the pair of linear combinations showing the strongest canonical correlation between combinations that were not correlated in the first set. Usually we can express quantitatively the relationship between the two sets.

The multivariable analysis has the following steps [11]:

a. Initial data – data processing and sorting in a suitable database.

b. Data cleaning – the removal of dataset samples that are inadequate or contains missing values.

c. Visual analysis – allows preliminary correlations and outlier detection.

d. Correlations - main factors in selecting variables by using methods such as MVA, PLS, multiple regressions.

e. Selection of variables – choosing a larger number of variables for a complete process, but also to have a correct statistical prediction and removing similar variables. This is done using correlation methods - in our case multiple regression.

f. Group analysis – helps to remove exceptional values (outliers), thus improving the prediction, and confirm if the prediction is indeed viable and valid.

Table 1

Variables used for analysis models and statistical prediction
Variabilele folosite pentru elaborarea modelelor matematice şi analiză statistică

Code / *Cod*	Signification / *Definiţie*	Minimum / maximum values *Valorile minime / maxime*
Independent variables / *Variabile independente*		
Al_2O_3 (%)	Mass proportion of Al_2O_3 / *Proporţia masică de* Al_2O_3	20/80
ZrO_2* (%)	Mass proportion of ZrO_2 stabilized with 8% Y_2O_3 / *Proporţia masică de* ZrO_2 *stabilizat cu 8%* Y_2O_3	20/80
Temp. (°C)	Heat treatment temperature / *Temperatura de tratament termic*	1400/1600
Soaking time (min.)	Soaking time / *Palierul de menţinere la temperatura maximă*	180/360
ZnO (%)	Mass proportion of ZnO / *Proporţia masică de ZnO*	0/2
CuO (%)	Mass proportion of CuO / *Proporţia masică de CuO*	0/2
MnO_2 (%)	Mass proportion of MnO_2 / *Proporţia masică de* MnO_2	0/2
TiO_2 (%)	Mass proportion of TiO_2 / *Proporţia masică de* TiO_2	0/2
Dependent variables / *Variabile dependente*		
Shrinkage (%)	Shrinkage / *Contracţie*	40.62/63.96
ρ_a (g/cm^3)	Apparent density / *Densitatea aparentă*	2.8/5.38
ρ_r (%)	Relative density / *Densitatea relativă*	67.72/99.71
Abs. (%)	Absorption / *Absorbţia*	0.09/15.51
Pd (%)	Open porosity / *Porozitatea deschisă*	0.97/50.77
Rc (MPa)	Compressive strength / *Rezistenţa mecanică la compresiune*	69.41/152.35
E (GPa)	Elasticity, Young's modulus / *Modulul de elasticitate Young*	0.13/1.95

2. Results and discussion

All the experimental data, presented in paper *Yttria-stabilized ZrO_2-Al_2O_3 dense composite ceramics. Obtaining and characterization* (published in same journal number) [12] were considered for present multivariable analysis. After initial data processing and sorting, from above mentioned results there have been removed 3 sets of data. Thus, there were developed seven mathematical models that can be further used for prediction (after validation [13,14]) with 15 variables (8 independent variables and 7 dependent variables) and 77 experimental data sets. The considered variables are presented in Table 1.

Logically, it has to remove the variable ZrO_2 content because it is reversed proportional with variable Al_2O_3 content, but by doing so it was noticed a decrease of confidence intervals and R^2 values of statistical models of ceramic and mechanical properties between 3 and 9%.

To create these statistical models it has been used the Statgraphics Centurion XVI program [15].

Due to relatively low number of data sets and independent variables (proportion of Al_2O_3 and ZrO_2, sintering treatment conditions - temperature and soaking time; oxide additives) have limited opportunities to improve these statistical models.

A good criterion for choosing a model configuration is to have simultaneously R^2 biggest coefficient and smallest prediction errors.

2.1. Mathematical models

Shrinkage model

Dependent variable: Shrinkage (%)

Independent variables: Al_2O_3 (%) ZrO_2* (%) Temp. (°C), Soaking time (min.) ZnO (%), CuO (%), MnO_2 (%) TiO_2 (%)

Table 2

Mathematical factors for shrinkage model / *Factorii matematici pentru modelul matematic al contracției*

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	-5.70223	5.09581	0.2671
Al_2O_3 (%)	0.103075	0.0262134	0.0002
ZrO_2* (%)	0.011895	0.0278055	0.6709
Temp. (°C)	0.0359412	0.00297234	0,0000
Soaking time (min.) *Palierul de tratament termic (min.)*	0.0124736	0.00259626	0.0000
ZnO (%)	2.21619	0.343207	0.0000
CuO (%)	-.323974	0.343207	0.0000
MnO_2 (%)	-0.822973	0.348988	0.0212
TiO_2 (%)	-3.35831	0.348988	0.0000

R^2 = 91.0223%

Estimated standard error = 1.79564

Average error modulus = 1.33611

Shrinkage model equation is:

Shrinkage (%) = -5.70223 + 0.103075·Al_2O_3 (%) + 0.011865·ZrO_2* (%) + 0.0359412·Temp. (°C) + 0.0124736·Soaking time (min.) + 2.21619·ZnO (%) -3.23974·CuO (%) -0.822973·MnO_2 (%) -3.35831·TiO_2 (%)

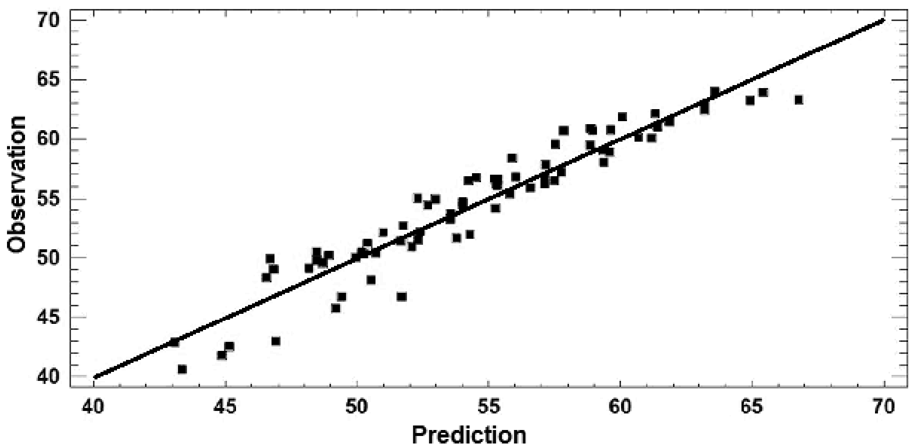

Fig. 1 - Shrinkage model / *Modelul matematic pentru contracție.*

Apparent density model

Dependent variable: ρ_a = (g/cm^3)

Independent variables: Al_2O_3 (%) ZrO_2* (%) Temp. (°C), Soaking time (min.) ZnO (%), CuO (%), MnO_2 (%) TiO_2 (%)

Table 3

Mathematical factors for apparent density model / *Factorii matematici pentru modelul matematic al densităţii aparente*

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	2.82402	0.402741	0.0000
Al_2O_3 (%)	-0.0306131	0.00207174	0.0000
ZrO_2* (%)	-0.00257457	0.00219757	0.2455
Temp. (°C)	0.00181384	0.000234915	0.0000
Soaking time (min.) *Palierul de tratament termic (min.)*	0.000893947	0.000205192	0.0000
ZnO (%)	0.232388	0.0271249	0.0000
CuO (%)	0.0839509	0.0271249	0.0029
MnO_2 (%)	0.170099	0.0275818	0.0000
TiO_2 (%)	-0.0172341	0.0275818	0.5342

R^2 = 96.0923%
Estimated standard error = 0.141916
Average error modulus = 0.110841

Apparent density model equation is:

ρ_a (g/cm3) = 2.82402 -0.0306131·Al_2O_3 (%) - 0.00257457·ZrO_2* (%) + 0.00181384·Temp. (°C) + 0.000893947·Soaking time (min.) + 0.232388·ZnO (%) + 0.0839509·CuO (%) + 0.170099·MnO_2 (%) - 0.0172341·TiO_2 (%)

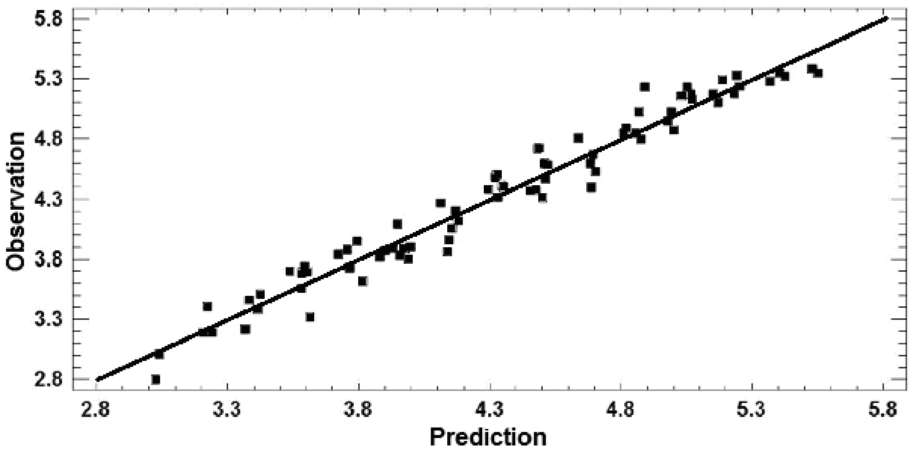

Fig. 2 - The apparent density model / *Modelul matematic pentru densitatea aparentă.*

Relative density model

Dependent variable: ρ_r (%)

Independent variables: Al_2O_3 (%) ZrO_2* (%) Temp. (°C), Soaking time (min.) ZnO (%), CuO (%), MnO_2 (%) TiO_2 (%)

Table 4

Mathematical factors for relative density model / *Factorii matematici pentru modelul matematic al densităţii relative*

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	39.6808	8.40648	0.0000
Al_2O_3 (%)	-0.252222	0.0432439	0.0000
ZrO_2* (%)	-0.0800385	0.0458703	0.0855
Temp. (°C)	0.0382213	0.00490342	0.0000
Soaking time (min.) *Palierul de tratament termic (min.)*	0.0201588	0.00428301	0.0000
ZnO (%)	5.38649	0.566183	0.0000
CuO (%)	1.90587	0.566183	0.0013
MnO_2 (%)	4.17262	0.575719	0.0000
TiO_2 (%)	-0.253052	0.575719	0.6617

R^2 = 85.4859%
Estimated standard error = 2.96224
Average error modulus = 2.3375
The relative density model equation is

ρ_r (%) = 39,6808 - 0,252222·Al_2O_3 (%) – 0,0800385·ZrO_2* (%) + 0,0382213·Temp. (°C) + 0,0201588·Soaking time (min.) + 5,38649·ZnO (%) + 1,90587·CuO (%) + 4,17262·MnO_2 (%) – 0,253052·TiO_2 (%)

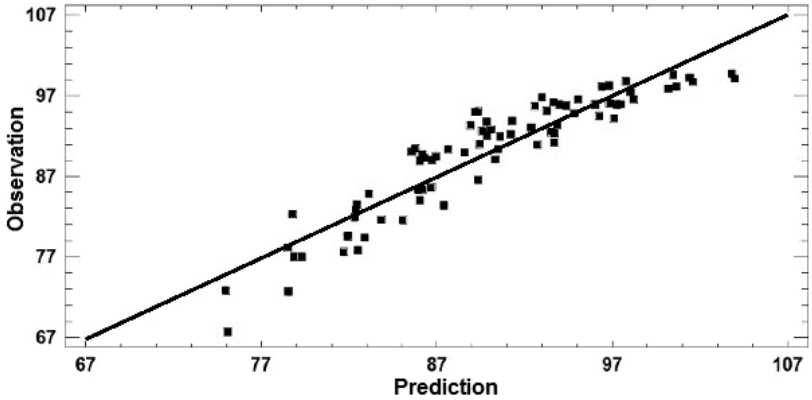

Fig. 3 - The relative density model / *Modelul matematic pentru densitatea relativă.*

Absorption model
Dependent variable: Abs. (%)
Independent variables: Al_2O_3 (%) ZrO_2* (%) Temp. (°C), Soaking time (min.) ZnO (%), CuO (%), MnO_2 (%) TiO_2 (%)

Table 5

Mathematical factors for absorption model / *Factorii matematici pentru modelul matematic al absorbției*

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	30.8884	5.71924	0.0000
Al_2O_3 (%)	0.0940236	0.024204	0.0021
ZrO_2* (%)	0.0273965	0.0312073	0.3831
Temp. (°C)	-0.0208035	0.00333598	0.0000
Soaking time (min.) *Palierul de tratament termic (min.)*	-0.00717832	0.00291389	0.0163
ZnO (%)	-1.50282	0.385196	0.0002
CuO (%)	-0.311885	0.385196	0.4209
MnO_2 (%)	-1.18837	0.391683	0.0034
TiO_2 (%)	0.781959	0.391683	0.0499

R^2 = 68,2132%
Estimated standard error = 2.01532
Average error modulus = 1.4882
Absorption model equation is:
Abs. (%) = 30.8884 + 0.0940236·Al_2O_3 (%) + 0.0273965·ZrO_2* (%) - 0.0208035·Temp. (°C) – 0.00717832·Soaking time (min.) - 1.50282·ZnO (%) - 0.311885·CuO (%) - 1.18837·MnO_2 (%) + 0.781959·TiO_2 (%)

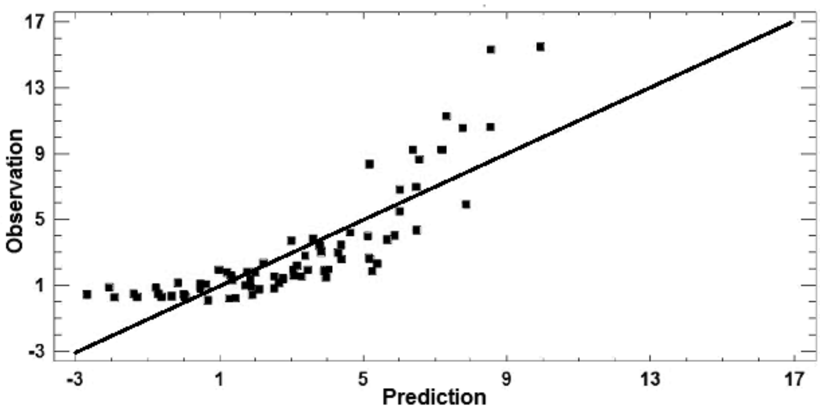

Fig. 4. Absorption model / *Modelul matematic pentru absorbție*.

Open porosity model

Dependent variable: Pd (%)

Independent variables: Al_2O_3 (%) ZrO_2* (%) Temp. (°C), Soaking time (min.) ZnO (%), CuO (%), MnO_2 (%) TiO_2 (%)

Table 6

Mathematical factors for open porosity model / *Factorii matematici pentru modelul matematic al porozităţii deschise*

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	118.241	20.2479	0.0000
Al_2O_3 (%)	0.261719	0.104157	0.0144
ZrO_2* (%)	0.0617015	0.110483	0.5784
Temp. (°C)	-0.0763003	0.0118104	0.0000
Soaking time (min.) *Palierul de tratament termic (min.)*	-0.0277221	0.0103161	0.0090
ZnO (%)	-4.351	1.36371	0.0021
CuO (%)	0.794937	1.36371	0.5619
MnO_2 (%)	-3.38545	1.38668	0.0172
TiO_2 (%)	3.56522	1.38668	0.0123

R^2= 66,6501 %

Estimated standard error = 7.13485

Average error modulus = 4.93547

Open porosity model equation is:

Pd (%) = 118.241 + 0.261719·Al_2O_3 (%) + 0.0617015·ZrO_2* (%) - 0.0763003·Temp. (°C) - 0.0277221·Soaking time (min.) - 4.35·ZnO (%) + 0.794937·CuO (%) - 3.38545·MnO_2 (%) + 3.56522·TiO_2 (%)

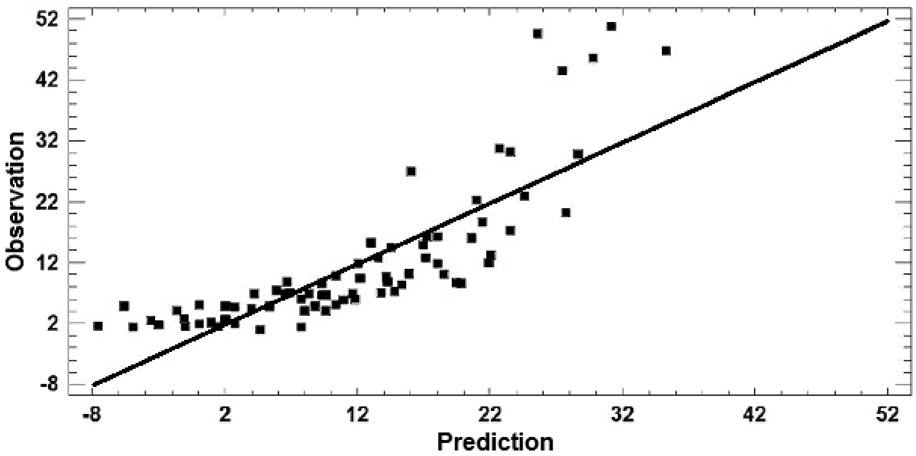

Fig. 5 - Open porosity model / *Modelul matematic pentru porozitatea deschisă.*

Compressive strength model

Dependent variable: Rc (MPa)

Independent variables: Al_2O_3 (%) ZrO_2* (%) Temp. (°C), Soaking time (min.) ZnO (%), CuO (%), MnO_2 (%) TiO_2 (%)

Table 7

Mathematical factors for compressive strength model

Factorii matematici pentru modelul matematic al rezistenţei mecanice la compresiune

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	-32.9786	41.8797	0.4337
Al_2O_3 (%)	-0.525366	0.215434	0.0174
ZrO_2* (%)	-0.35714	0.228518	0.1227
Temp. (°C)	0.122417	0.024428	0.0000
Soaking time (min.) *Palierul de tratament termic (min.)*	-0.012758	0.0213372	0.5519
ZnO (%)	7.33005	2.82063	0.0115
CuO (%)	-4.43557	2.82063	0.1205
MnO_2 (%)	4.45182		0.1253
TiO_2 (%)	-8.01118	2.86814	0.0068

R^2 = 55,0366 %

Estimated standard error = 14.7574

Average error modulus = 11.8973

Compressive strength model equation is:

Rc (MPa) = -32.9786 - 0.525366·Al_2O_3 (%) - 0.35714·ZrO2* (%) + 0.122417·Temp. (°C) - 0.012758·Soaking time (min.) + 7.33005·ZnO (%) - 4.43557·CuO (%) + 4.45182·MnO_2 (%) - 8.01118·TiO_2 (%)

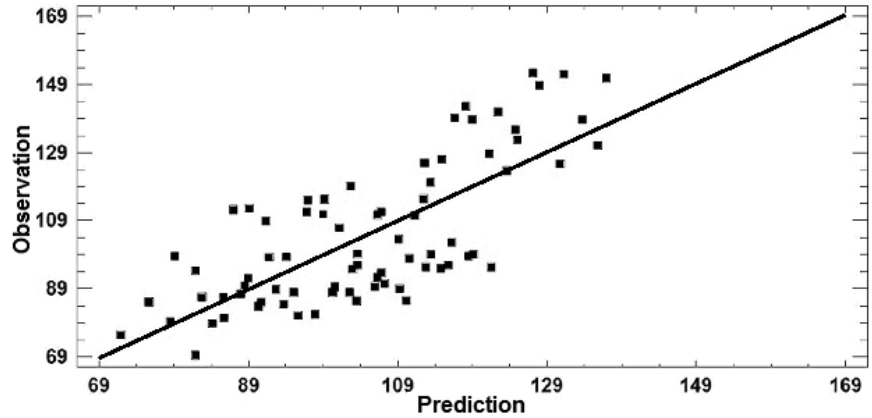

Fig. 6 - Model for compressive strength / *Modelul matematic pentru rezistența mecanică la compresiune.*

Improved compressive strength model

Dependent variable: Rc (MPa)

Independent variables: Al_2O_3 (%), ZrO_2* (%), Temp. (°C), Soaking time (min.), ZnO (%), CuO (%), MnO_2 (%), TiO_2 (%), Cont. (%), ρ_a (g/cm^3), ρ_r (%), Abs. (%), Pd (%)

Table 8

Mathematical factors for improved compressive strength model

Factorii matematici pentru modelul matematic îmbunătățit al rezistenței mecanice la compresiune

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	-67.584	83.5778	0.4218
Al_2O_3 (%)	0.143129	0.578371	0.8053
ZrO_2* (%)	-0.547321	0.217475	0.0144
Temp. (°C)	0.127883	0.0454646	0.0065
Soaking time (min.) *Palierul de tratament termic (min.)*	-0.00105289	0.0249974	0.9665
ZnO (%)	12.2481	4.40662	0.0072
CuO (%)	2.40802	4.60937	0.6032
MnO_2 (%)	12.0866	3.87691	0.0027
TiO_2 (%)	-4.14578	4.41551	0.3514
Shrinkage (%) Contracție (%)	1.25915	0.956357	0.1927
ρ_a (g/cm^3)	59.9319	24.9881	0.0194
ρ_r (%)	-3.76637	1.32296	0.0060
Abs. (%)	1.47288	2.25492	0.5160
Pd (%)	-0.198794	0.586612	0.7358

R^2 = 64,6937 %

Estimated standard error = 13.5859

Average error modulus = 10.3082

Equation improved compressive strength model is

Rc (MPa) = 67584 + 0.143129·Al_2O_3 (%) - 0.547321·ZrO_2* (%) + 0.127883·Temp. (°C) - 0.00105289·Soaking time (min.) + 12.2481·ZnO (%) + 2.40802·CuO (%) + 12.0866·MnO_2 (%) - 4.14578·TiO_2 (%) + 1.25915·Shrinkage (%) + 59.9319·ρ_a (g/cm^3) - 3.76637·ρ_r (%) + 1.472·Abs. (%) -0.198794·Pd (%)

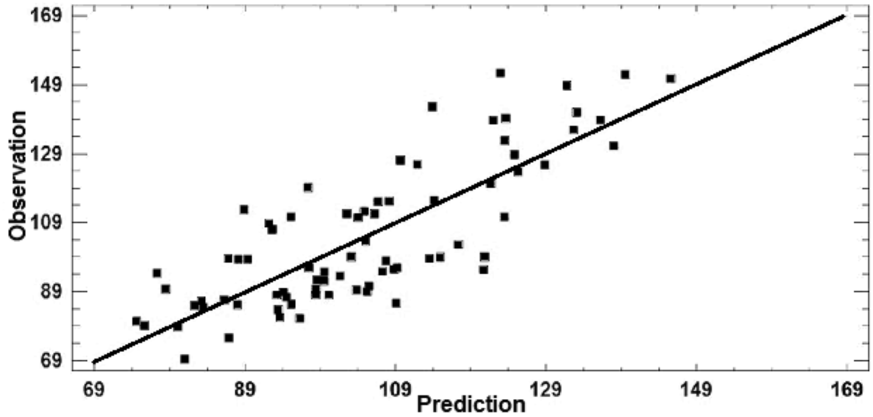

Fig. 7 - The improved compressive strength / *Modelul matematic îmbunătățit pentru rezistența mecanică la compresiune.*

By introducing the ceramic properties as independent variables will get a higher quality model. It requires a more rigorous control of these properties to improve compressive strength.

Elasticity model
Dependent variable: E (GPa)
Independent variables: Al_2O_3 (%) ZrO_2* (%) Temp. (°C), Soaking time (min.) ZnO (%), CuO (%), MnO_2 (%) TiO_2 (%)

Table 9

Mathematical factors for elasticity model / *Factorii matematici pentru modelul matematic al elasticității*

Parameter / *Parametru*	Estimation / *Estimare*	Standard Error / *Eroarea Standard*	P value / *Valoarea P*
CONSTANT	7.06183	0.922835	0.0000
Al_2O_3 (%)	-0.00929966	0.00474717	0.0542
ZrO_2* (%)	-0.00310011	0.00503548	0.5402
Temp. (°C)	-0.00354445	0.000538281	0.0000
Soaking time (min.) *Palierul de tratament termic (min.)*	-0.000129835	0.000470174	0.7833
ZnO (%)	0.0542653	0.0621537	0.3857
CuO (%)	-0.366047	0.0621537	0.0000
MnO_2 (%)	0.105133	0.0632005	0.1008
TiO_2 (%)	-0.432201	0.0632005	0.0000

R^2 = 75,0661 %
Estimated standard error = 0.325184
Average error modulus = 0.244117

Elasticity model equation is:
E (GPa) = 7.06183 - 0.00929966·Al_2O_3 (%) - 0.00310011·ZrO_2* (%) -0.00354445·Temp. (°C) - 0.000129835·Soaking time (min.) + 0.0542653·ZnO (%) -0.366047·CuO (%) + 0.105133·MnO_2 (%) - 0.432201·TiO_2 (%)

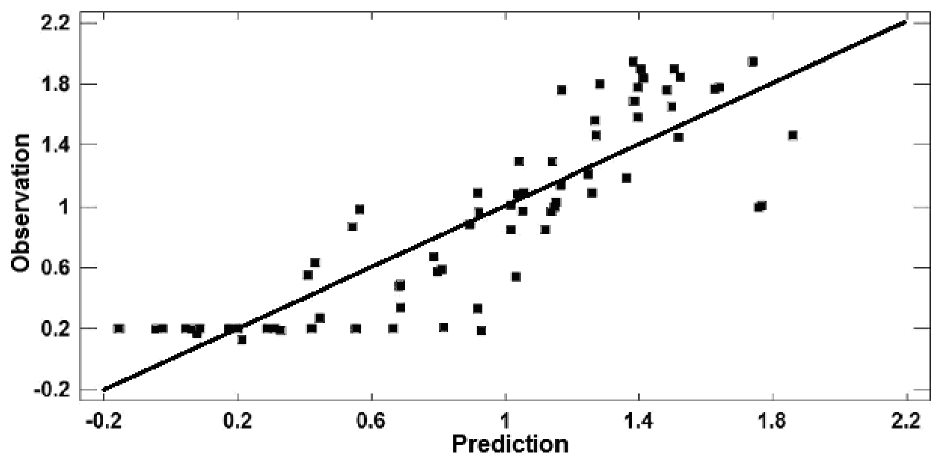

Fig. 8 - Elasticity model / *Modelul matematic pentru modulul de elasticitate.*

Groups analysis

For this analysis it were considered 77 complete data sets, the clustering method was the nearest neighbor and the considered method of calculating the distance was Euclidean quadratic - it calculates the sum of squares of differences of corresponding values.

This procedure created a single group of the 77 data sets. Groups are sets of data with similar characteristics. To form groups, the procedure starts with each set in a separate group, and then it combines the two groups that are closest, then calculating the distance between groups. The closest two groups are then combined into one; this step is repeated until obtaining a single group. The oxide additives variables were removed due to the fact that they were added in very low amounts.

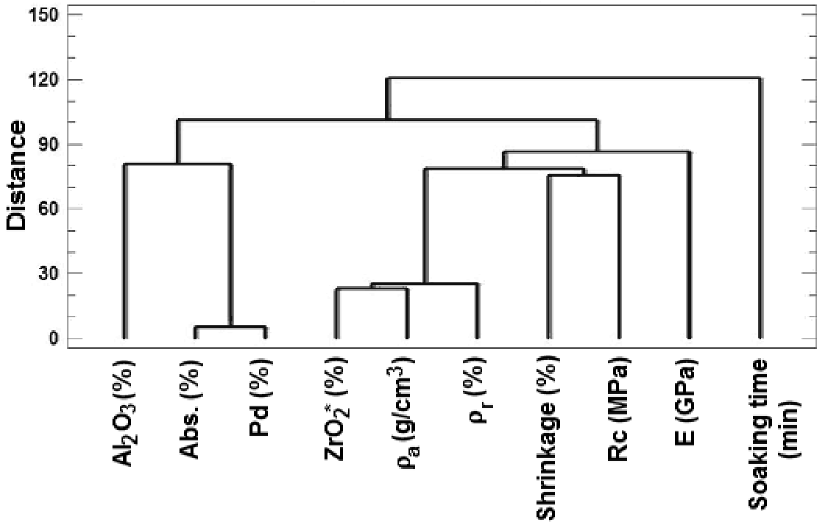

Fig. 9 - Dendrogram / *Dendrogramă.*

From this groups analysis of analysis, it can be seen that all the variables are grouped according to experimental data [12].

3. Conclusions

From done researches we can conclude the following:

- Statistical models designed for ceramic properties are superior to those for mechanical properties.
- The introduction of ceramic properties as independent variables we get a higher quality model for compressive strength.
- Statistical models created are in accordance with existing assertions in this area - are viable, but also offers new insights, are interesting from the point of view of optimizing the compositions as well as the specific properties of ceramics.

ACKNOWLEDGEMENTS

The experimental results from this study were obtained with the support of Ministry of Labor, Family and Social Protection through the Sectoral Operational Programme Human Resources Development 2007-2013, no. POSDRU/88/1.5/S/60203.

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