Utilizarea învățării automate pentru generarea computerizată a diagramelor de echilibru termic fazal din date de difractometrie de raze X

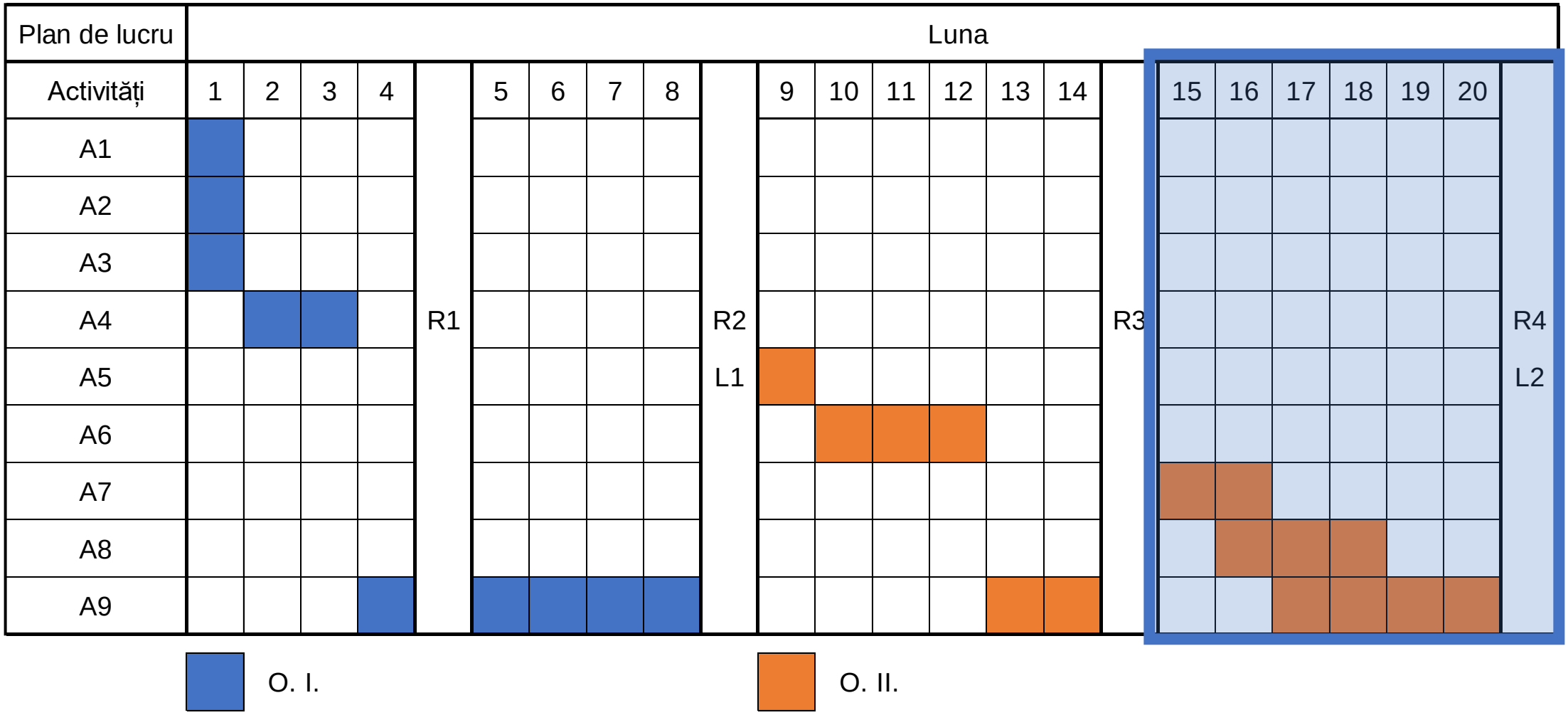
Raport 4: Analiză comparativă a prelucrării manuale și a utilizării învățării automate pentru prelucrarea difractogramelor de raze X în sistemul oxidic $BiFeO_3$–$EuFeO_3$

AOSR-TEAMS 2023-2024

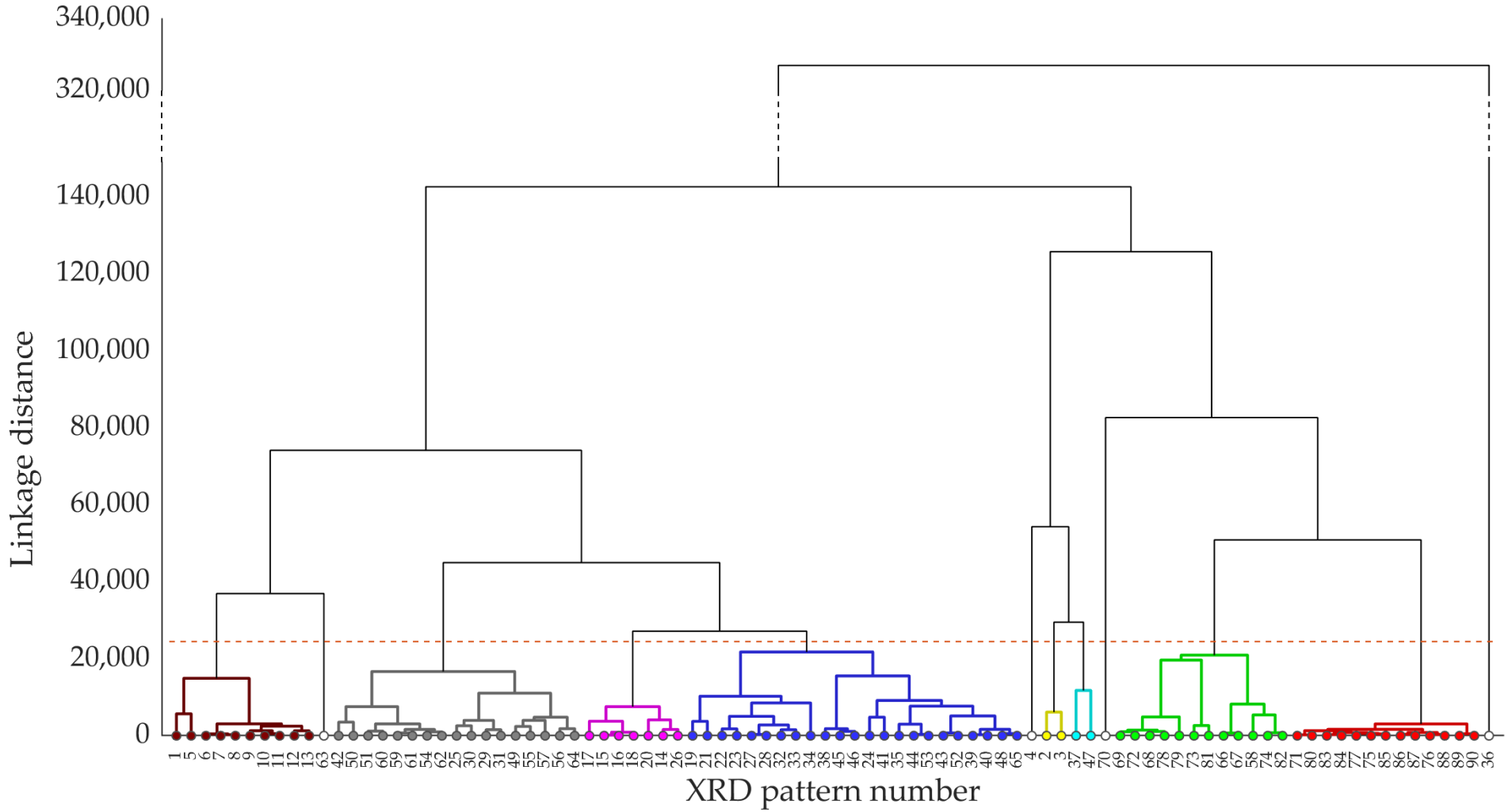
Conducător de proiect: Vasile–Adrian SURDU

Membru: Romuald GYŐRGY

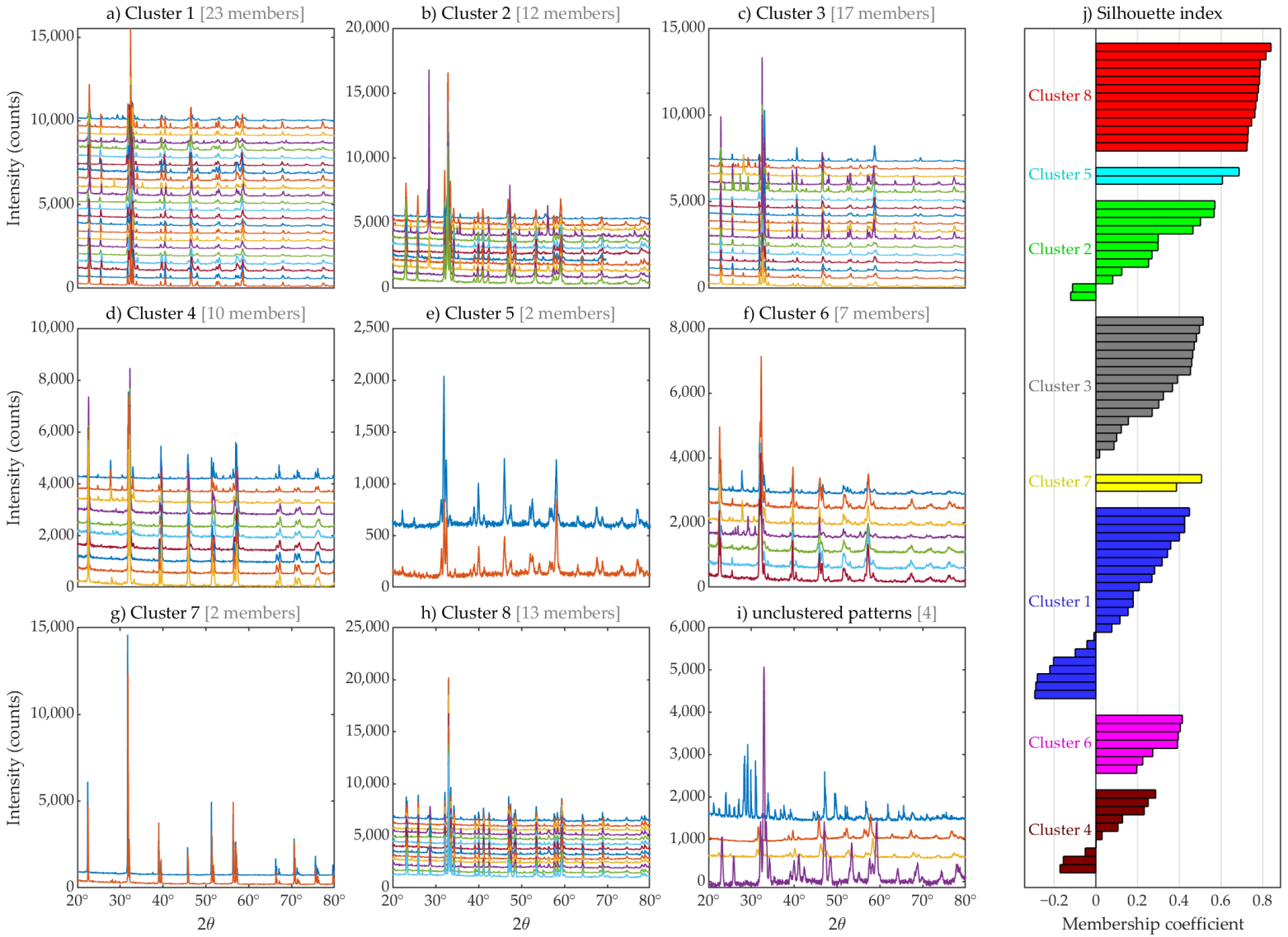
Calendarul activităților



Activitatea 6. Analiza difractogramelor de raze X [...] utilizând învățarea automată



Activitatea 6. Analiza difractogramelor de raze X [...] utilizând învățarea automată



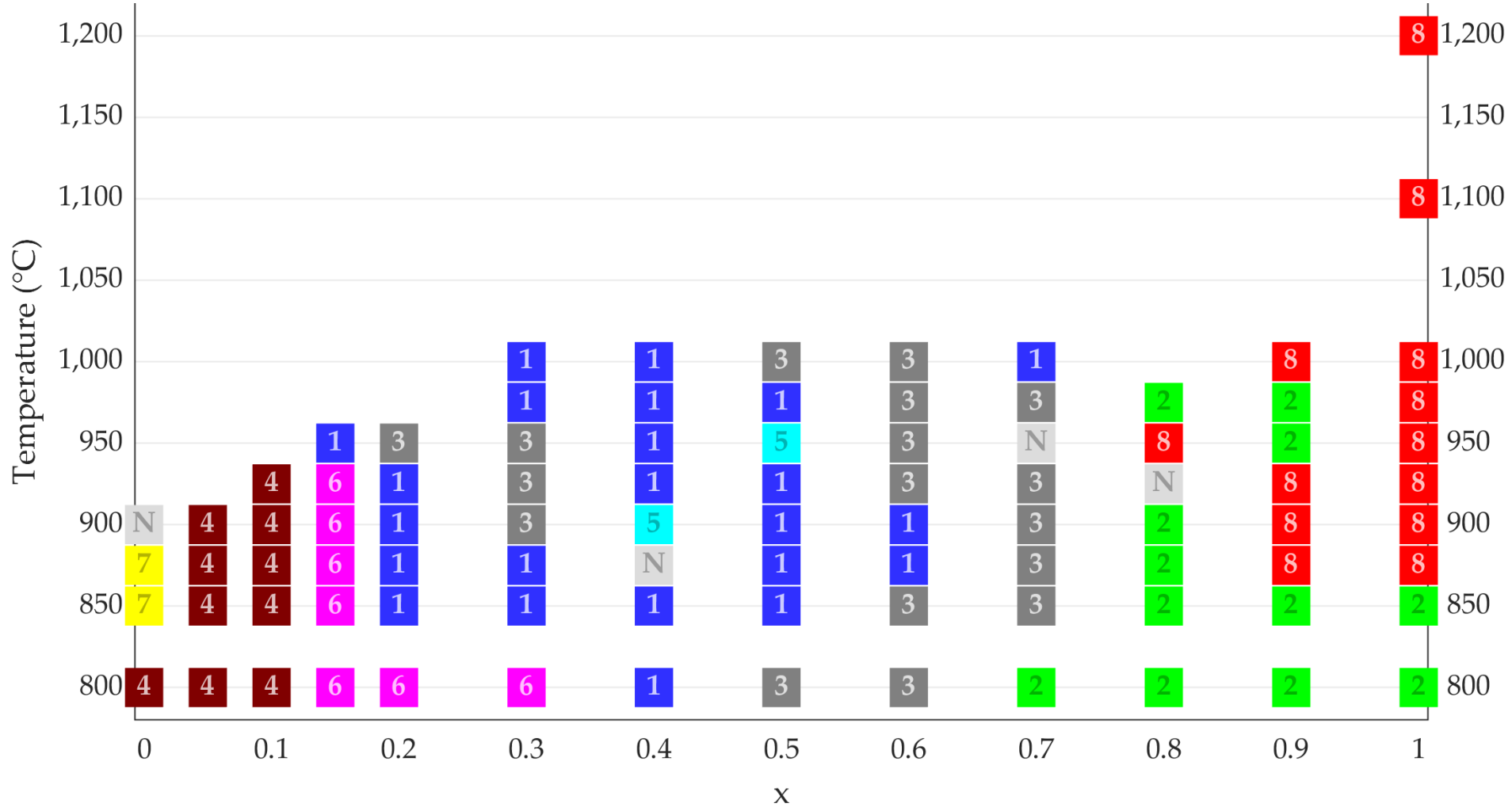
Pentru generarea rezultatelor a fost necesară conectarea software (HighScore Plus – MATLAB)

```
1)  fs = dir(fullfile('input', '*.xrdml'));
2)  fileName = string({fs.name});
3)  % infer Eu percentage (0 < x < 1)
4)  xpercent = str2double(regexp(fileName, '(?<=Eu)\d+', 'match', 'once'));
5)  locus = ismissing(xpercent);
6)  oneminusx = str2double(regexp(fileName, '(?<=Bi)\d+', 'match', 'once'));
7)  assert(isequal(locus, ismissing(oneminusx)))
8)  assert(all(xpercent(~locus) + oneminusx(~locus) == 100))
9)  % infer Eu percentage (x=0 OR x=1)
10) BiEu = regexp(fileName, 'Bi|Eu', 'match’);
11) assert(all(cellfun(@numel, BiEu(~locus))==2))
12) missingX = xpercent(locus);
13) missingX([BiEu{locus}]=="Bi") = 0;
```

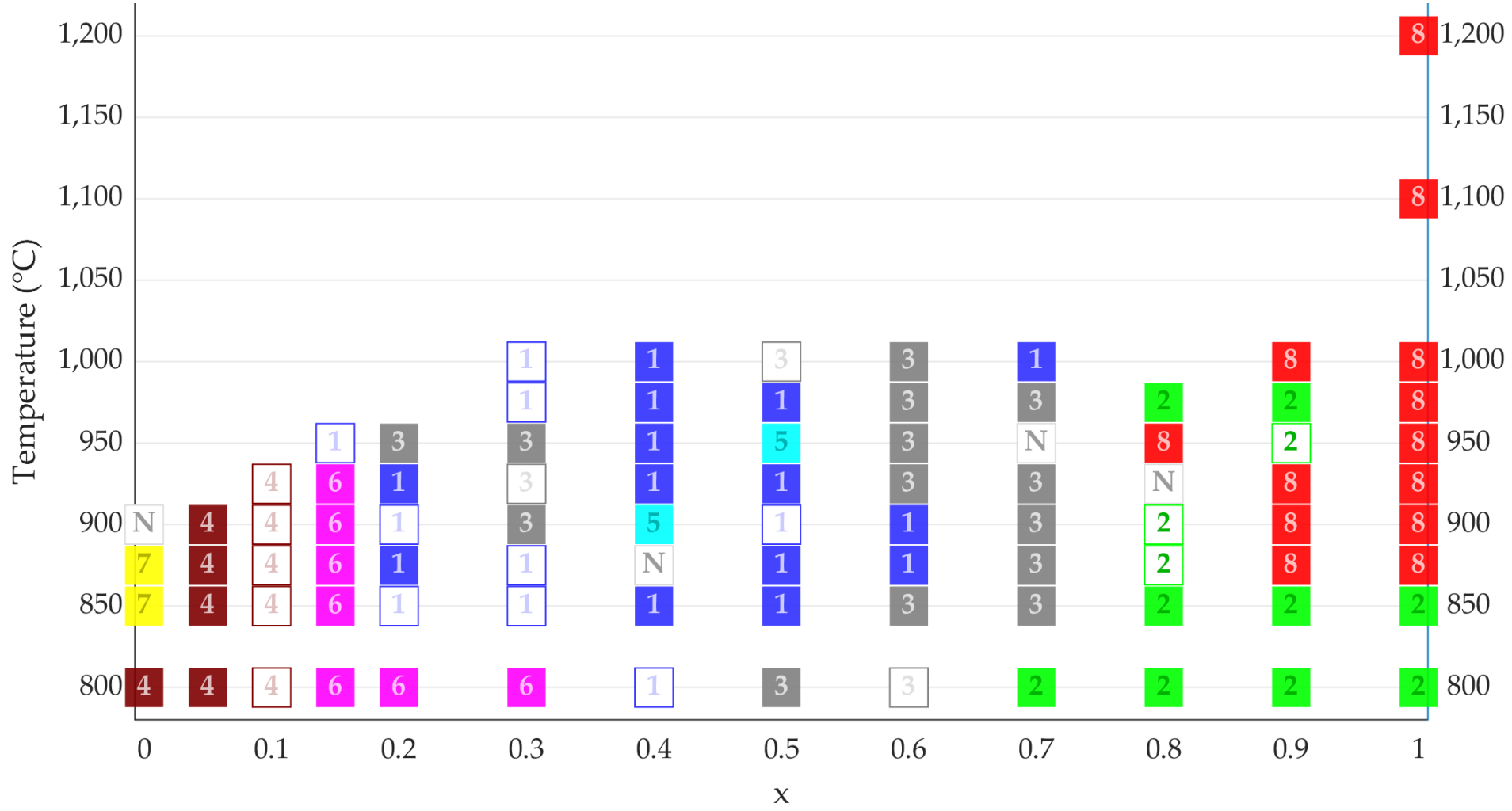
Pentru generarea rezultatelor a fost necesară conectarea software (HighScore Plus – MATLAB)

```
14) missingX([BiEu{locus}]=="Eu") = 100;
15) assert(nnz(ismissing(missingX))==0)
16) xpercent(locus) = missingX;
17) % infer temperature values
18) str = regexp(fileName, '(?<=_)\w+?(?=(_2h)?\.xrdml$)', 'match', 'once’);
19) temperature = str2double(str);
20) temperature(str=="presinterizat") = 800;
21) assert(nnz(ismissing(temperature))==0)
22) % order by Eu percentage, then temperature
23) [XT, idx] = sortrows([xpercent(:), temperature(:)]);
24) fileName = fileName(idx);
25) path = fs(1).folder;
26) save(fullfile('+pfig','data.mat'), '-append', 'path', 'fileName', 'XT');
```

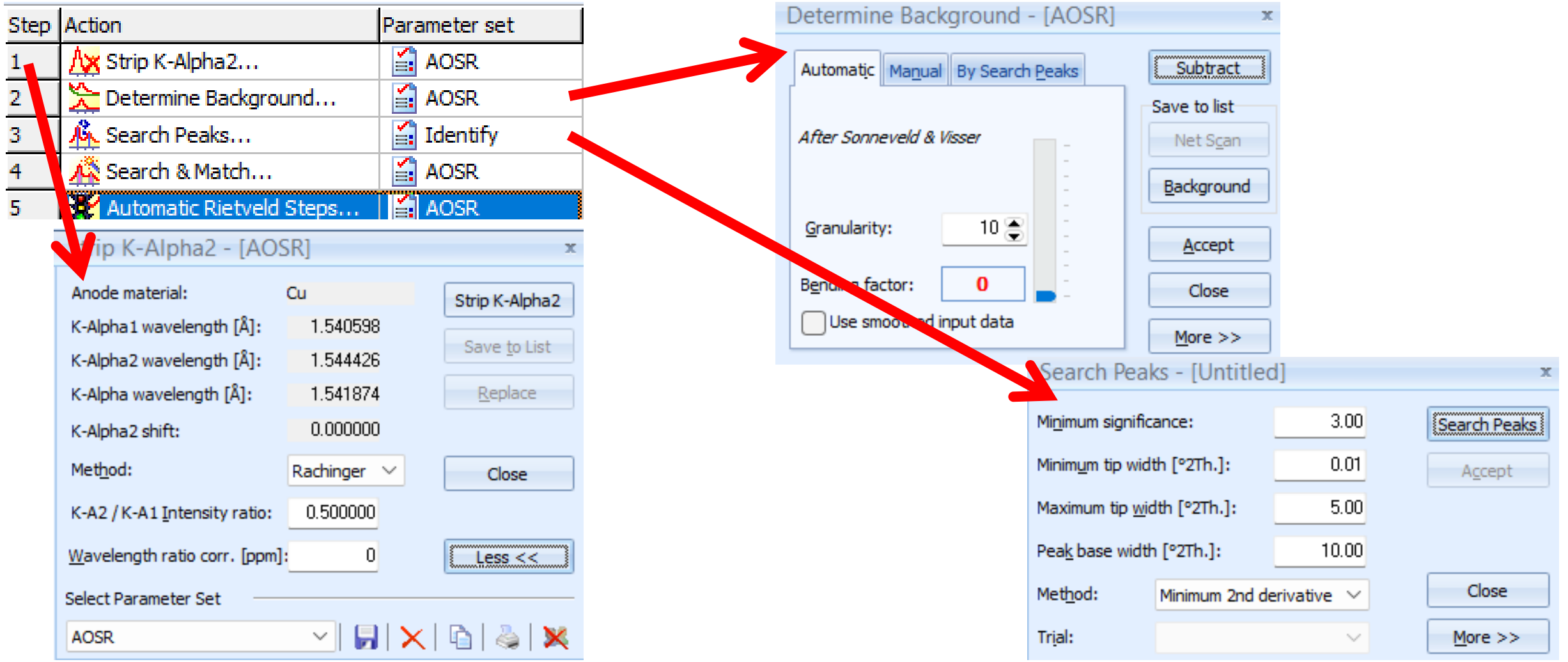
Activitatea 8. Stabilirea relațiilor de echilibru termic fazal din date de difractometrie de raze X



Activitatea 8. Stabilirea relațiilor de echilibru termic fazal din date de difractometrie de raze X

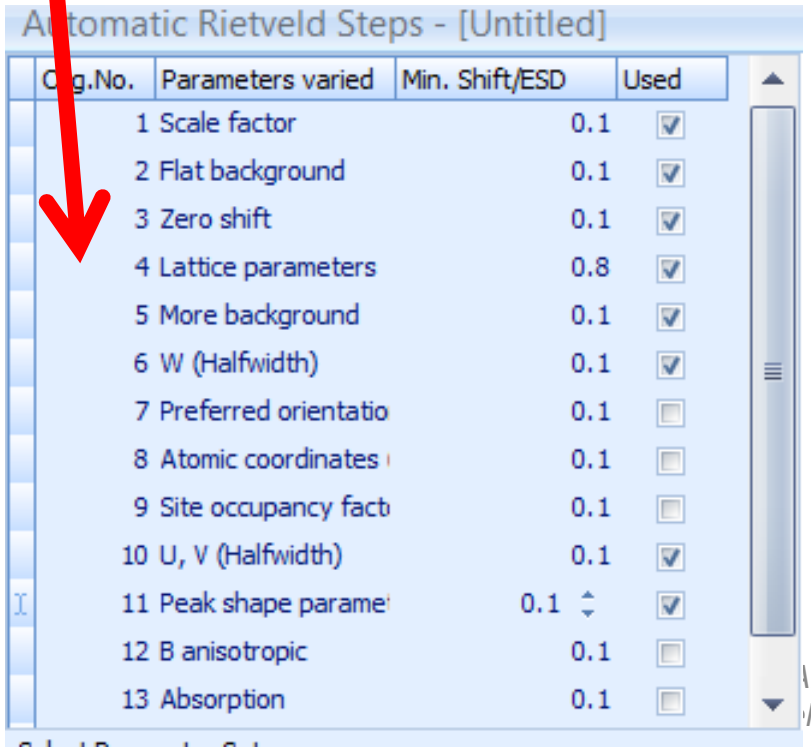


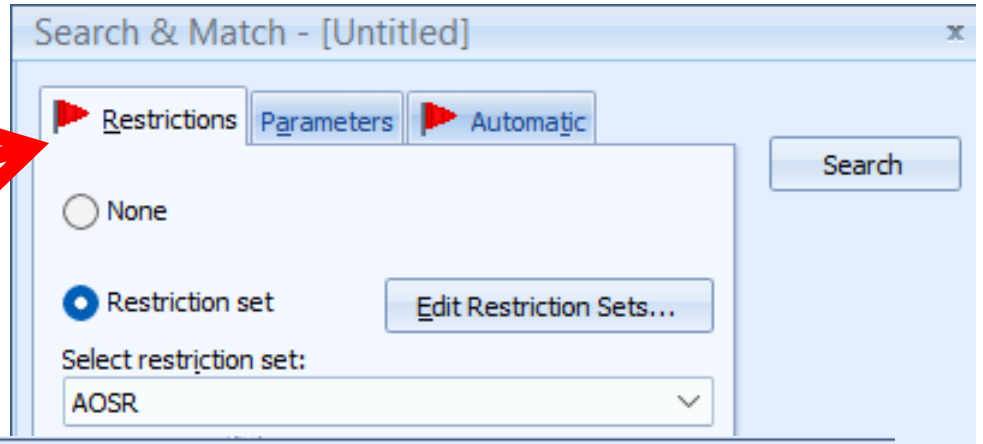
Activitatea 7. Identificarea și cuantificarea fazelor utilizând metoda Rietveld: prelucrare automată

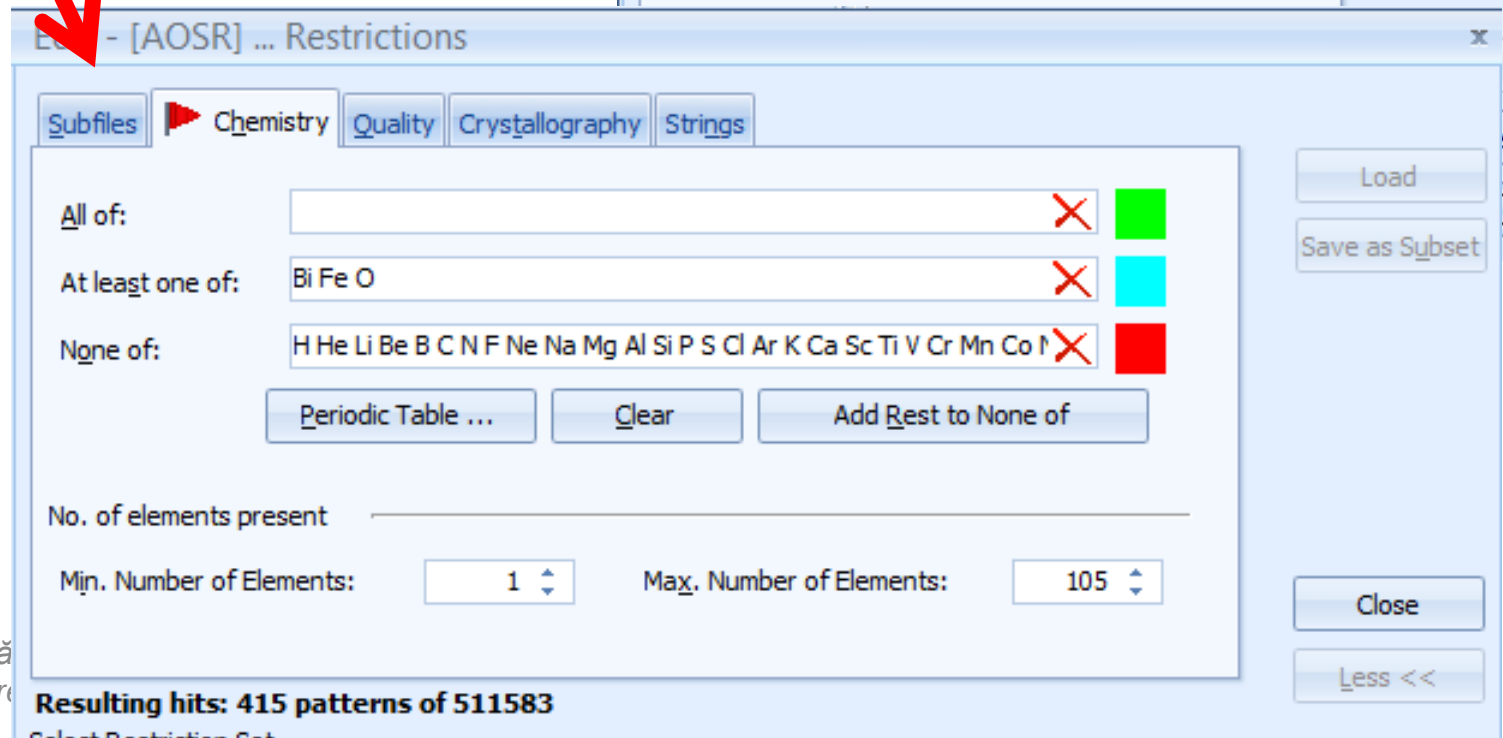


Activitatea 7. Identificarea și cuantificarea fazelor utilizând metoda Rietveld: prelucrare automată

Step	Action	Parameter set
1	Strip K-Alpha2...	AOSR
2	Determine Background...	AOSR
3	Search Peaks...	Identify
4	Search & Match...	AOSR
5	Automatic Rietveld Steps...	AOSR







Activitatea 7. Identificarea și cuantificarea fazelor utilizând metoda Rietveld: rezultate

XRD pattern number	x	T(°C)	(Bi,Eu)FeO_3-r	Sillenite-type	Mullite-type	Bi_2O_3	$Bi_2Eu_2Fe_4O_{12}$	(Bi,Eu)FeO_3-o (Pbnm)	$Eu_3Fe_5O_{12}$	Eu_2O_3	Fe_2O_3
1	0	800	84.7%	15.3%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
2	0	850	100.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
3	0	875	99.0%	0.9%	0.1%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
4	0	900	0.0%	0.5%	99.5%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
5	0.05	800	80.6%	19.4%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
6	0.05	850	100.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
7	0.05	875	100.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
8	0.05	900	99.7%	0.0%	0.0%	0.3%	0.0%	0.0%	0.0%	0.0%	0.0%
9	0.1	800	80.2%	16.5%	1.9%	0.0%	1.4%	0.0%	0.0%	0.0%	0.0%
10	0.1	850	98.4%	0.4%	0.4%	0.0%	0.8%	0.0%	0.0%	0.0%	0.0%
11	0.1	875	99.2%	0.5%	0.0%	0.0%	0.3%	0.0%	0.0%	0.0%	0.0%
12	0.1	900	100.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
13	0.1	925	99.4%	0.6%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%

Activitatea 7. Identificarea și cuantificarea fazelor utilizând metoda Rietveld: rezultate

XRD pattern number	x	T(°C)	(Bi,Eu)FeO_3-r	Sillenite-type	Mullite-type	Bi_2O_3	$Bi_2Eu_2Fe_4O_{12}$	(Bi,Eu)FeO_3-o (Pbnm)	$Eu_3Fe_5O_{12}$	Eu_2O_3	Fe_2O_3
14	0.15	800	53.0%	14.1%	1.5%	0.0%	31.4%	0.0%	0.0%	0.0%	0.0%
15	0.15	850	49.9%	0.0%	0.0%	0.0%	50.1%	0.0%	0.0%	0.0%	0.0%
16	0.15	875	81.0%	0.0%	0.0%	0.0%	19.0%	0.0%	0.0%	0.0%	0.0%
17	0.15	900	83.2%	0.0%	0.0%	0.0%	16.8%	0.0%	0.0%	0.0%	0.0%
18	0.15	925	64.0%	0.0%	1.8%	0.0%	34.2%	0.0%	0.0%	0.0%	0.0%
19	0.15	950	32.1%	0.5%	4.0%	0.0%	63.4%	0.0%	0.0%	0.0%	0.0%
20	0.2	800	38.9%	8.8%	1.5%	0.0%	50.8%	0.0%	0.0%	0.0%	0.0%
21	0.2	850	31.2%	0.3%	1.3%	0.0%	67.2%	0.0%	0.0%	0.0%	0.0%
22	0.2	875	67.7%	0.0%	0.0%	0.0%	32.3%	0.0%	0.0%	0.0%	0.0%
23	0.2	900	65.1%	0.0%	0.0%	0.0%	34.9%	0.0%	0.0%	0.0%	0.0%
24	0.2	925	44.1%	0.0%	4.3%	0.0%	51.6%	0.0%	0.0%	0.0%	0.0%
25	0.2	950	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
26	0.3	800	43.1%	0.0%	17.7%	0.0%	39.2%	0.0%	0.0%	0.0%	0.0%

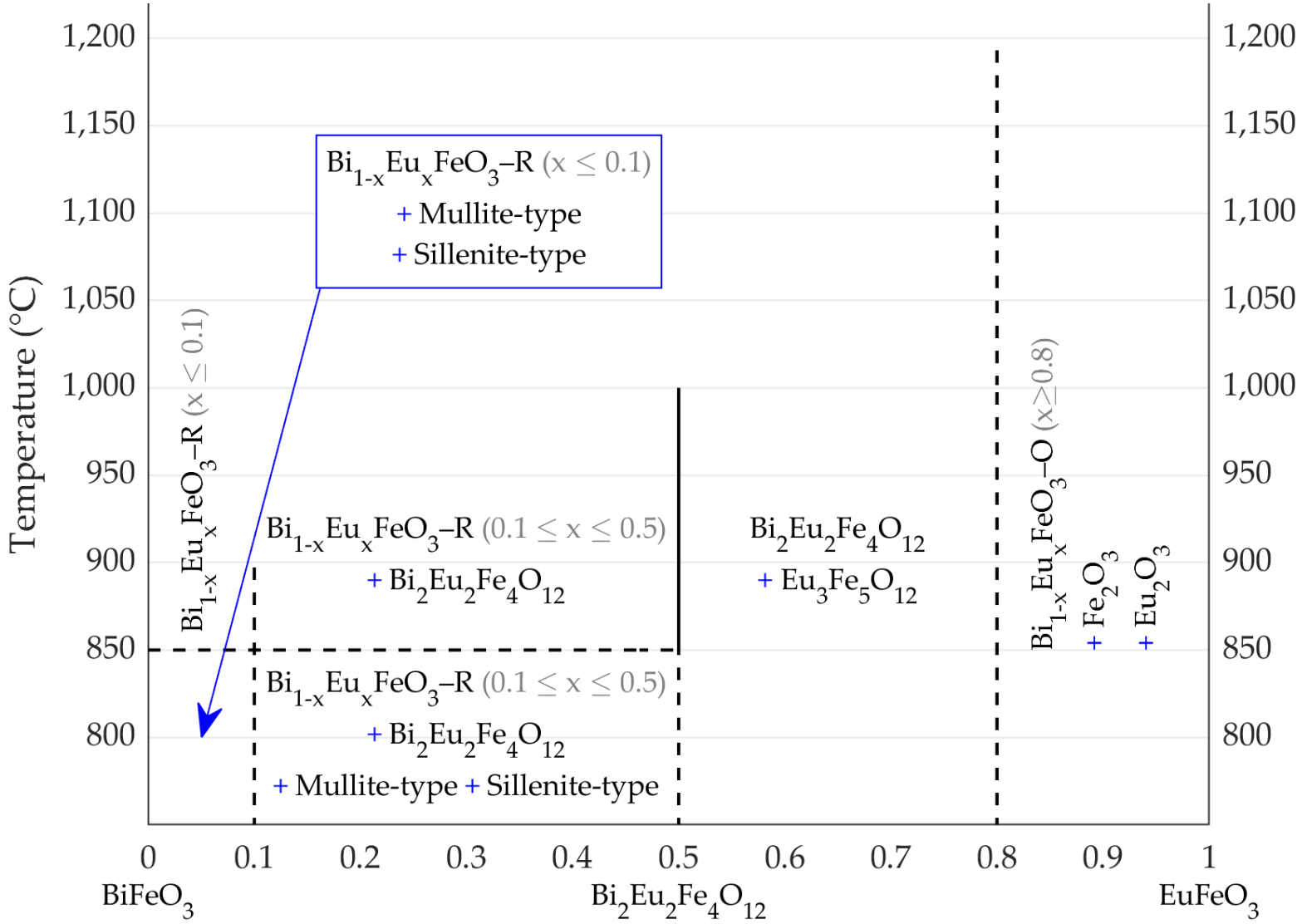
Activitatea 7. Identificarea și cuantificarea fazelor utilizând metoda Rietveld: rezultate

XRD pattern number	x	T(°C)	(Bi,Eu)FeO_3-r	Sillenite-type	Mullite-type	Bi_2O_3	$Bi_2Eu_2Fe_4O_{12}$	(Bi,Eu)FeO_3-o (Pbnm)	$Eu_3Fe_5O_{12}$	Eu_2O_3	Fe_2O_3
27	0.3	850	13.7%	0.0%	0.0%	0.0%	86.3%	0.0%	0.0%	0.0%	0.0%
28	0.3	875	9.7%	0.0%	0.0%	0.0%	90.3%	0.0%	0.0%	0.0%	0.0%
29	0.3	900	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
30	0.3	925	0.0%	2.5%	8.1%	0.0%	89.4%	0.0%	0.0%	0.0%	0.0%
31	0.3	950	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
32	0.3	975	12.2%	0.5%	4.3%	0.0%	83.0%	0.0%	0.0%	0.0%	0.0%
33	0.3	1000	16.0%	0.0%	0.0%	0.0%	84.0%	0.0%	0.0%	0.0%	0.0%
34	0.4	800	21.8%	0.3%	2.1%	0.0%	75.8%	0.0%	0.0%	0.0%	0.0%
35	0.4	850	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
37	0.4	900	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
38	0.4	925	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
39	0.4	950	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
40	0.4	975	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%

Activitatea 7. Identificarea și cuantificarea fazelor utilizând metoda Rietveld: rezultate

XRD pattern number	x	T(°C)	(Bi,Eu)FeO_3-r	Sillenite-type	Mullite-type	Bi_2O_3	$Bi_2Eu_2Fe_4O_{12}$	(Bi,Eu)FeO_3-o (Pbnm)	$Eu_3Fe_5O_{12}$	Eu_2O_3	Fe_2O_3
38	0.4	925	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
39	0.4	950	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
40	0.4	975	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
41	0.4	1000	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
42	0.5	800	24.7%	0.0%	6.3%	0.0%	69.0%	0.0%	0.0%	0.0%	0.0%
43	0.5	850	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
44	0.5	875	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
45	0.5	900	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
46	0.5	925	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
48	0.5	975	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
49	0.5	1000	0.0%	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%
50	0.6	800	0.0%	1.1%	17.5%	0.0%	81.4%	0.0%	0.0%	0.0%	0.0%

Activitatea 8. Stabilirea relațiilor de echilibru termic fazal din date de difractometrie de raze X



Sumarul activităților realizate

- ✓A fost explorată utilizarea metodelor de învățare automată utilizate pentru prelucrarea difractogramelor de raze X. Studiul a avut în vedere sistematizarea etapelor necesare pentru a fi parcurse, a parametrilor utilizați și a corelației între parametrii de analiză și rezultatul urmărit.
- ✓A fost utilizată învățarea automată pentru clasificarea difractogramelor de raze X obținute pentru domenii de compoziție nestudiate (până acum) ale unor sisteme oxidice de interes practic
- ✓Am utilizat secvențe de instrucțiuni de calculator pentru automatizarea prelucrării difractogramelor de raze X.
- ✓Au fost determinate relațiile de echilibru termic fazal în sistemul oxidic $BiFeO_3$–$EuFeO_3$.

Indicatorii proiectului

Lucrare 1 (recenzie) cu tema: Algoritmi de învățare automată în prelucrarea difractogramelor de raze X: principii și aplicații – F.I. min. 0.6

F.I. = 2.5 (Q1)

Open Access Review

X-ray Diffraction Data Analysis by Machine Learning Methods—A Review

by **Vasile-Adrian Surdu** [1,2] and **Romuald Győrgy** [2,3,*]

[1] Department of Science and Engineering of Oxide Materials and Nanomaterials, Faculty of Chemical Engineering and Biotechnologies, National University of Science and Technology Politehnica Bucharest, Gheorghe Polizu 1-7, 011061 Bucharest, Romania

[2] Academy of Romanian Scientists, Ilfov 3, 050044 Bucharest, Romania

[3] Department of Chemical and Biochemical Engineering, Faculty of Chemical Engineering and Biotechnologies, National University of Science and Technology Politehnica Bucharest, Gheorghe Polizu 1-7, 011061 Bucharest, Romania

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Indicatorii proiectului

Lucrare 2 (articol) cu tema: Relații de echilibru termic fazal în sistemul oxidic $BiFeO_3$–$EuFeO_3$ determinate din date de difracție de raze X prin algoritmi de învățare automată – F.I. min. 0.6

F.I. = 3.1 (Q2)

Open Access Article

Phase Relations in the Pseudo-Binary $BiFeO_3$–$EuFeO_3$ System in the Subsolidus Region Derived from X-Ray Diffraction Data—A Machine Learning Approach

by **Vasile-Adrian Surdu** [1,2] and **Romuald Győrgy** [2,3,*]

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