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on Fundamental and Applied Aspects of
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CONTENT

<i>Volume II</i>	
<i>Organic Physical Chemistry</i>	553
<i>Material Science</i>	583
<i>Physical Chemistry of Condensed Phases, Solid State and Fluids</i>	709
<i>Macromolecular Physical Chemistry</i>	731
<i>Environmental Protection, Forensic Sciences, Geophysical Chemistry,</i>	761
<i>Radiochemistry, Nuclear Chemistry</i>	
<i>Phase Boundaries, Colloids, Liquid Crystals, Surface-Active Substances</i>	857
<i>Complex Compounds</i>	879
<i>General Physical Chemistry</i>	907
<i>Pharmaceutical Physical Chemistry</i>	921
<i>Education, History</i>	991
<i>Food Physical Chemistry</i>	1005
<i>Physico-Chemical Analysis</i>	1039
<i>INDEX</i>	1057

CARBON CRYOGEL AS AN ADSORBENT FOR REMOVAL OF DRUGS AND PESTICIDES FROM WATER

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ABSTRACT

Carbon cryogel (CC), obtained by pyrolysis of resorcinol–formaldehyde cryogels, was chemically modified and used for adsorption of drugs and pesticides from water. FTIR analysis and point of zero charge determination showed that applied chemical treatments affect the surface chemistry of carbon cryogel by changing the acidity of the surface as well as the nature and amount of surface oxygen groups. Generally, the chemical modification has no influence on drugs adsorption, while both chemical treatments increase the adsorption of pesticides onto carbon cryogel surface.

INTRODUCTION

Carbon cryogels (CC) represent a class of carbon materials that draw attention due to their developed and controllable mesoporosity. Since adsorption characteristics of carbon materials depend not only on surface porosity, but also on surface chemistry, adsorption properties of CC, obtained by pyrolysis of resorcinol–formaldehyde cryogels [1, 2], was altered by applying chemical modification with various agents. Alteration of surface chemistry, i.e. amount and nature of surface oxygen groups, can change the wettability of CC surfaces, and consequently make it more hydrophilic and suitable for the adsorption of relatively low molecular weight and polar compounds. On the other hand, functional groups may increase diffusion resistance and reduce the accessibility and affinity of CC surfaces for organic chemicals. Therefore, finding the optimal method of surface modification is crucial for improving CC adsorption properties toward the selected pesticides and drugs.

EXPERIMENTAL

CC was modified by 4 M KOH or 4 M HNO₃. 5 g of pristine CC was suspended in 25 mL of reagent and stirred at 80°C for 4 h. Then, the suspension was filtered and washed with deionized water until neutral pH. The obtained materials were labeled CC/KOH and CC/HNO₃, which refers to samples treated with potassium hydroxide and nitric acid, respectively.

The point of zero charge (PZC) was determined by mass titration, by placing various amounts (0.05, 0.1, 0.5, 1 and 10% by weight) of material in 10 cm³ of 0.1 M KCl solution. The beakers were shaken overnight, and the limiting pH value was taken as the PZC.

Fourier Transform Infrared Spectroscopy (FTIR) measurements were used for characterization of functional groups. FTIR spectra were recorded in the range from 400–4000cm⁻¹ on Bomem MB-Series, Hartmann Braun.

Adsorption of selected pesticides (imidacloprid (Im) and acetamiprid (Ac)) and drugs (sulfamethoxazole (S), diclofenac (D) and erythromycin (E)) by unmodified and modified CC samples were performed from aqueous solutions, in the batch system at 25°C. Initial concentration per each compound was 20 mg/dm³. 0.05 g of CC samples were immersed in 50 cm³ of pesticides/drugs solution and constantly shaken for three hours. To determine the optimal pH value of the aqueous solution, the effect of pH on CC samples adsorption capacities was studied. The concentration of selected pesticides and drugs in the solution were measured by high-performance liquid chromatography-tandem mass spectrometry (LTQ XL, Thermo Scientific).

RESULTS AND DISCUSSION

FTIR spectra, for both unmodified and modified CC samples are shown in Fig. 1a. The wide band between 3300 and 3700 cm⁻¹ is assignable to the stretching vibrations of O-H bond, whereas the bands around 2922 and 2850 cm⁻¹ are to be ascribed to asymmetric and symmetric C-H stretching vibrations, respectively. The peak at 1637 cm⁻¹ is assigned to OH bending vibration [3, 4], while the band at 1620 cm⁻¹ can be attributed to the aromatic C=C bond. The band at 1384 cm⁻¹ can be attributed to carboxyl O-H deformation vibrations. Bands in the range 1500-1000 cm⁻¹ can be assigned to the C-O stretching and O-H bending vibrations, which imply the existence of hydroxyl, ester or ether groups. For all samples, FTIR spectra are similar and show a presence of oxygen groups on CC surface. Applied chemical treatments alter the surface chemistry, especially in the case of HNO₃ treatment, which enhances the amount of surface oxygen groups. These changes in the surface chemistry, induced by chemical modification, also reflect on the values of the pH_{PZC} (Fig. 1b.).

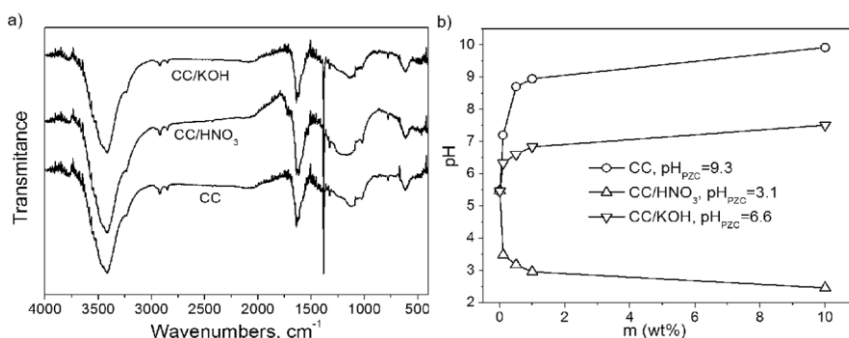


Figure 1. Characterization of CC samples: a) FTIR spectra and b) PZC

Adsorption properties of CC samples tested through the pesticides and drugs adsorption are presented in Fig. 2. Although the pesticides adsorption equilibrium onto modified CC samples has not been achieved after 180 minutes, applied chemical treatments improve pesticide adsorption (Fig. 2a). The influence of chemical modification on drugs adsorption on CC (Fig. 2b) surface primarily depends on particular drug. Chemical modification has no influence on adsorption of sulfamethoxazole, while decreases the adsorption of erythromycin. Adsorption of diclofenac is slightly increased by chemical modification with KOH, while HNO₃ modification of CC decreases diclofenac adsorption.

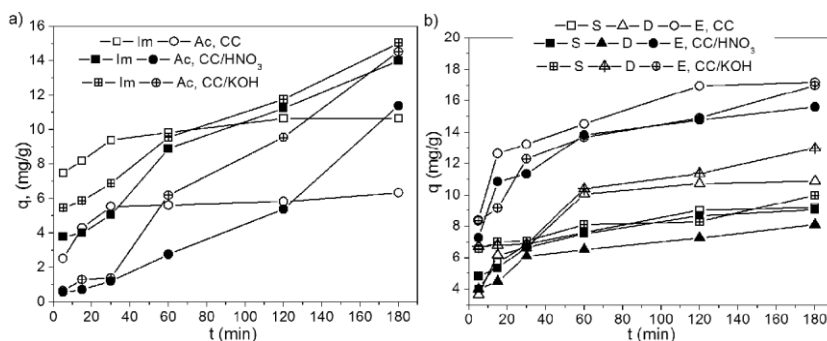


Figure 2. Amount of the pesticides (a) and drugs (b) adsorbed at the surface of CC samples

Effect of initial pH on the pesticides and drugs adsorption on unmodified and modified CC samples is shown in Fig. 3. It was found that optimal pH value of the aqueous solution for pesticides adsorption was pH=4 on CC and CC/HNO₃, while for CC/KOH maximal adsorption was achieved at pH=2 (Fig. 3a). For all tested materials optimal pH value of the aqueous solution for drugs adsorption was pH=8 (Fig. 3b).

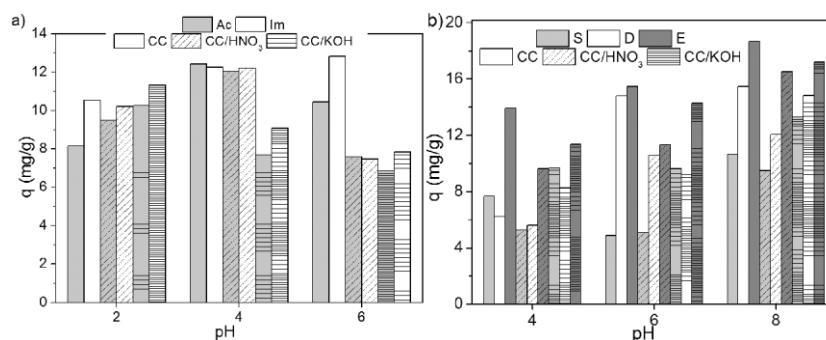


Figure 3. Effect of initial pH on the pesticides (a) and drugs (b) adsorption on unmodified and modified CC samples

CONCLUSION

Applied chemical treatments affect the surface chemistry of carbon cryogel by changing the acidity of the surface as well as the nature and amount of surface oxygen groups. Chemical modification generally has no influence, or deteriorate the drugs adsorption onto carbon cryogel. On the other hand, both chemical treatments increase the adsorption of pesticides onto carbon cryogel surface.

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Abazović N.	229	Antić M.	395
	677		959
Adnađević B.	625	Antić V.	515
	805	Antić-Stanković J.	471
	943	Antonijević Nikolić	
Agbaba D.	97	M.	895
	923		471
	931		903
	1041	Arandić D.	515
Ahmetović S.	237	Arbuzov A.B.	209
Ahrenkiel S. P.	391	Aroguz A.	749
Ajduković M.	185	Arsenijević A.N.	149
	371	Aștefanei D.	629
Aleksić J.	559	Avdeev G.	213
	563	Avdović E.	575
	567	Avdović E.H.	93
Aleksić M.	363		113
	379	Azizoğlu A.	129
	383	Babić B.	817
Amić D.	59	Badnjević A.	665
Amić A.	59	Bajuk Bogdanović D.	367
	121	Balvanović R.	825
	93	Banković P.	185
Amosov A.A.	63		225
Anđelković D.	777		371
	841	Baranac-Stojanović	
	1015	M.	559
			563
Anđelković M	317		567
Anđelković T.	777		
	841	Barbov B.	10
	1015	Bartolić D.	523
Anić S.	337	Beljanski M.V.	527
	349		543
Anitas E.	629	Bera O.	685
	633	Berdon K.	967
	637	Berezin S.S.	145
Antić B.	617	Beškoski V.	499
		Beškoski V.P.	737

	963	Brdarić T.	785
Birgaoanu D.	629	Brothers E.N.	84
Biswas D.J.	81	Bubanja I.N.	321
Blagojević B.	455		341
Blagojević Filipović J.P.	571	Budinski-Simendić J.	499
Blagojević S.M.	871		715
	875	Bulanov E.N.	749
	987	Burian T.	609
Blagojević S.N.	321	Cakić M.	763
	875	Cakić S.	891
	1011	Čamdžjija Z.	749
Blazhevaska-Gilev J.	585	Camuka H.	527
Blekkan E.A.	241	Çarıkcı S,	159
Blokhina A.G.	609	Cebzan A.	129
Bojić A.	593	Čeklić S.	101
	597	Cervellati R.	515
	681	Chen L.	337
Bojić D.	597	Cherepanova S.V:	159
	681	Cherepanova S.V.	201
Boldyrev A.E.	947	Ciganović J.	205
Bondžić A.	503	Ciraj-Bjelac O.	85
	511	Čirić-Marjanović G.	515
	705	Civiš S.	399
Bondžić B.P.	463	Čolović M.	763
	483		443
	563		503
Borisov G.A.	613		507
Borisov V.A	209	Čomor M.	511
Borodin A. O.	221		229
Božić B.	325	Cricenti A.	677
Božović P.	515		82
Brandin J.	241	Crişan D.	507
Brankov M.	547	Csépes-Ruzicska L.J.	621
	1019	Čučulović A.	217
Branković M	841	Čučulović R.	821
Brborić J.	379	Čudina O.	821
			379

	951	Djoković N.	923
Ćujić M.	797	Djordjević D.B.	149
	845	Djuran M.I.	149
	849	Djuričić T.	387
Culicov O.	825	Dobričić V.	379
Čupić Ž.	296		951
	317	Dobrokhotova Zh.V.	887
	337		899
	349	Dobrosavljević M.	931
Ćurlin M.	781	Dodevski V.	641
Cvetković T.	777	Dojčinović B.P.	617
Daljević Lj.	987	Dolcet P.	159
Damian F.	629	Donnard M.	881
	633	Đokić M.	845
Damian G.	629	Đoković N.	105
	633	Đolić M.	797
	325		849
Damjanović J.	661	Đorđević I.	153
Danilov P.A.	491	Đorđević M.	845
Danilović B.	757	Đorđević M.J.	455
Dapčević A.	527		459
Delić N.	475	Đorđević M.R.	455
Devečerski A.	809	Đorović J.	121
	813		575
Đikanović D.	531	Dostál J.	763
	867	Dostanić J.	161
Dimić D.	113		245
	479		249
	575	Dragić M.	535
			539
Dimitrić Marković J.M.	113	Dragičević V.	547
	479		1019
	575	Dragović R.	845
Dimitrijević A.	825	Dragović S.	797
	859		845
	863		849
Djerdj I.	159	Drakulić D.	435

	535	Gagić Ž.	97
	539	Gaković B.	661
Dražić B.	895	Ganesan A.	923
	903	Gavrikov A.V.	899
Dražić J.	471	Gavrilović Lj.	241
Drmać F.	853	Georgijević J.	785
Dudzak K.	763	Gerasimov A.V.	947
Đurđić S.	1045	Gezović A.	407
Đurkić T.	817	Gianturco F.A.	117
Đurović S.	1011	Gojgić-Cvijović B.	499
Džambaski Z.	463	Gojgić-Cvijović G.	737
	483		963
	559	Gorjanović S.	109
Džunuzović E.S.	745		395
	757		415
Džunuzović J.V	745	Goronja J.	867
	753	Gorzowski M.T.	279
	757	Govedarica O.	715
Efimov N.N.	887	Grahovac Z.	1053
	899	Graovac S.	459
Egerić M.	789	Greco E.	337
	813	Grinvald I.I	719
Ellinghaus R.	159	Grković I.	435
Emel'yanenko V.N.	63		535
Erić N.	871		539
	875	Gross S.	159
Fábián I.	217	Grujić S.	837
Ferus M.	763	Gruševac Stojanović	
Fijan D.	711	I.	539
Filipović N.R.	881	Gulea M.	881
Földényi R.	773	Gusarov E.V.	63
Franich A.	149	Guševac Stojanović I.	435
Furrow S.D.	271		535
Gabrovska M.	197	Gyevi-Nagy L.	312
	653	Hadnađev-Kostić M.	245
	657	Halasi T.	993
Gadžurić S.	863		997

	1001		715
Hederić Ž.	421		749
Holmen A.	241	Janković M.M.	829
Horváth A.K.	294	Janković Mandić Lj.	797
Horváth D.	312		845
Horváth O.	773		849
Hülvely B.M.	217	Janković S.	701
Idakiev V.	197	Janković-Častvan I.	407
	213	Janošević Ležaić A.	383
Iepure G.	629		867
	633		983
Ilić A.S.	395	Jauković Z.	837
Ilić B.S.	463	Jędrusiak M.	279
	483	Jelić D.	701
Ilić J.	809	Jerala R.	46
Ilić T.	383	Jeremić S.	121
Ilyukhin A.B.	887	Jerosimić S.	909
	899	Jerosimić S.V.	117
Ionin A.A.	661	Jevtić S.	1045
Iost K.N.	205	Jevtić V.V.	93
	209	Jocić A.	859
Isakova A.	287		863
Ivanek O.	763	Jokanović S.	829
Ivanković A.	853	Jokić A.	245
Ivanov I.	197		665
	213	Joksimović K.	963
Ivanov K.	213	Joksović Lj.	121
Jakovljević D.	963	Jovanović D.	495
Jakovljević D.M.	737	Jovanović G.	575
Janjić G.	467	Jovanović J.	625
Janjić G.V.	149		805
	153		943
Janjić G.V.	881	Jovanović J.M.	313
Janković D.	971	Jovanovic M.	313
	975	Jovanović M.	97
	979	Jovanović M.	363
Janković M.	685	Jović M.	789

Jović V.	605	Knyazev A.V.	63
Jovičić M.	685		609
Jović-Jovičić N.	185		613
	225	Knyazeva S.S.	63
	371	Kocić G.	841
Jovović A.	47		1015
Jugović D.	407	Kocić H.	1015
Juha L.	763	Kodranov I.D.	617
Jurczakowski R.	279		793
Kačarević-Popović Z.	495	Kolar Anić Lj.	21
Kalagaev I.Yu.	719		296
Kalamković M.	993		317
	997		337
	1001		349
Kalamković S.	993	Kolarević A.	463
	997		483
	1001	Kolentsova E.	213
Kalijadis A.	817	Komarova E.S.	403
Kalska B.	705		411
Kalvachev Yu.	10	Konovalov B.	149
Kanazir S.	451	Konstantinović S.	1027
Kapetanović V.	363	Koroteev P.S.	887
Kapustin R.V.	719		899
Karakirova Y.	213	Kortz U.	18
Karljiković-Rajić K.	363	Koskin A.P.	221
	935	Kostić B.	653
Karnaukhov T.M.	201	Kostić I.	777
	645		841
	205		1015
Kenzhin R.M.	205	Kostić M.	593
Kesić Ž.	193		597
Khalid O.	159		681
Kılıç T.	129		649
Kiss E.	169	Kostić M.M.	649
Kiss V.	217	Kosyakov A.V.	145
Klar P.J.	159	Koturević B.	943
Klekota U.	705	Kovačević B.	109
Knižek A.	763	Kovačević K.	983

Kovacević O.	109	Lazić B.	649
Kovacheva D.	197	Lelet M.I.	609
Kragović M.	641	Lente G.	34
Kraljević T.	781	Lević Lj.B.	1007
Krashennnikova O.V.	613	Li C.	159
Kravić N.	547	Lješević M.	499
Kresović B.	1019	Lončarević B.	499
Krstić D.	421	Lončarević B.D.	737
	503	Lončarević D.	161
	507		249
	511		605
Krstić J.	495		653
Krstić J.	621	Luce M.	82
Krstić S.	641		507
Krūs M.	763	Lugonja N.	963
Kubelík P.	763	Lukić I.	193
Kuč D.M.	793	Lutsyk V.	67
Kudryashov S.I.	661	Maćešić S.	296
Kuljanin –Jakovljević J.	229		349
Kumrić K.	475		353
	809		357
	813	Macura S.	49
Kuzmanović M.M.	85	Magsumov T.	917
	133	Maksimov G.V.	527
Laban B.	665	Maksimović J.	325
	669		337
	705		673
Lahtela-Kakkonen M.	923	Maksimović T.V.	673
Lakočević M.	487	Maksimović V.	523
Lamovec J.	605	Malenović A.	867
Lantos E.	312	Maletić M.	817
Laušević M.	649	Malinović B.N.	387
	817	Maljković M.	543
	837	Manojlović D.D.	617
Lazarević Đ.	515		793
Lazarević-Pašti T.	669	Manojlović V.	313

Marinković D.	653	Mentus S.	367
	657		375
Marinković F.	689		407
	693	Mesarović J.	547
	697	Micić D.	1007
Marinović S.	185		1011
	225		1023
	371	Mičić S.	983
Marković B.	935	Mihajlović I.	785
Marković B.M.	745	Milanović Z.	971
	753		975
	757		979
Marković J.	833	Milenković D.	121
Marković M.M.	665	Milenković D.	93
Marković M.M.	669		575
Marković M.P.	225	Miletić A.	1031
Marković S.	125		1035
Marković S.B.	237		1053
Marković V.	421	Milikić J.	391
Marković V.M.	296		399
	317	Milisavić D.	701
Marković Z.	59	Milojković J.	641
	113	Milojković-Opsenica	
	121	D.	547
Martinović Bevanda		Milovanović B.	657
A.	781		909
	853	Milovanović D.	661
Martinović J.	435	Milovanović J.Z.	149
	535	Milovanović M.	909
	539	Milutinović S.	785
Martinović M.	853	Milutinović-Nikolić	
Masnikosa R.	1049	A.	185
Matić Bujagić I.	837		225
Matović Lj.	475		371
	809	Minčić M.	987
	813	Minin V.V.	887
Mažić M.	321	Mirković M.	475
			971

	975		333
Mishakov	201	Myrstad R.	241
Mishra P.K.	49	Najdanović S.	593
Mitić M.	1031		681
	1035	Najman S.	491
Mitić N.	543		891
Mitić S. S	1053	Nakarada Đ.	451
Mitić S.M.	491		479
	891		519
Mitić S.S.	1031	Nastasović A.B.	745
	1035		753
Mitić Ž.	491		757
	891	Nedeljković J.	161
Mitrić B.	387	Nedić Z.P	673
Mitrić M.	741	Nešović M.	871
Mitrović J.	593		875
	597	Nestorović S.	821
Mitrović N.	435	Nikolić Z.	601
	535	Nikolić G.M.	233
	539		555
Mojović M.	451		891
	487	Nikolić G.S.	891
	519	Nikolić K.	97
Mojović Z.	185		101
	371		105
Möller M	159		923
Molnár M.	773	Nikolić M.	1031
Momčilović M.D.	85		1035
Momić T.	669		1053
Mraković A.	789	Nikolić M.G.	555
Mrmošanin J.	1031	Nikolova D.	621
Mudrinić T.	185		653
	225		657
	371	Nikšić M.	955
Mutavdžić D.	523		959
Muzika F.	255	Novaković K.	287
	263		349

Novaković T.B.	677	Pašti I.	399
Novotortsev V.M.	887	Pastor F.T.	395
	899		415
Obradović D.	1041	Pavićević A.	451
Ognjanović M.	617		487
Oljačić S.	1041		519
Omanović Miklićanin		Pavlić B.	1011
E.	665	Pavličević J.	685
Omerašević M.	475	Pavlov J.	527
	809	Pavlović A.	1031
	813		1035
Onjia A.E.	753		1053
	833	Pavlović D.	777
	849	Pavlović M.D.	527
Opsenica D.	547	Pavlović S.	653
Orlik M.	279		657
Osipov A.A.	947	Pavun L.	867
Ostojčić S.	109		983
	1007	Pecev-Marinković E.	1031
	1023		1035
Ostrovskii N	177		1053
Ősz K.	217	Pecić S.	955
Otoničar M.	741		959
Otto S.	32	Pejić B.	649
Over H.	159	Pejić N.	867
Özer Z.	129		983
Pagnacco M.C.	325	Pejin B.	519
	337	Pekala K.	279
	375	Pergal M.M.	793
Panić S.	169	Pergal M.V.	617
Pantić M.	955		793
	959		1045
Papan J.	161	Perić M.	971
Parac Vogt T.	503		975
Parmon V.	177		979
Pašalić S.	641	Perić M.I.	853
Pascal R.	20	Perić-Grujić A.	909

Perović M.	451	967
Peruško D.	661	1041
Peteović D.	853	Poreba R. 685
Petković A.	825	Potkonjak N.I 329
Petković B.B.	793	345
	1045	Potočňák I. 113
Petković Benazzouz		Prodanović O. 801
M.	153	Prodanović R. 801
Petković M.	923	Prokić B. 451
Petković S.	805	Prokopijević M. 801
Petrova P.	213	Prosheva M. 585
Petrović Đ.	475	Protopopov F. 527
	975	Rac V. 237
	979	Radaković N. 821
Petrović J.	797	Radenković M. 833
	845	Radenović Č.N. 527
	849	Radinović K. 0.391
Petrović M.	593	Radojković V. 255
	597	Radosavljević A. 495
	681	Radotić K. 523
Petrović M.P.	955	531
	959	801
Petrović S.	605	Radović J. 479
Petrović S.M.	661	Radović M. 971
Pieňkos M.	141	975
Pieňkos M.	939	979
Pijović M.	785	Radović M.D. 593
Pirutko L.V.	205	597
Plyusnin P.E.	205	681
Popović G.	931	Radulović V. 363
	967	935
	1041	Raičević J. 47
Popović S.	495	Rajčić B. 913
Popović-Belić A.	451	1049
	487	Rajković A. 105
	519	Rajković S. 149
Popović-Nikolić M.	931	Rakić A. 399

	467		133
Rakočević Z.	495	Schlender P.	701
Ralević U.	665	Schmitz G.	271
Ranković D.	805	Schreiber I.	255
Ranković D.P.	85		263
	133		333
Rašić Mišić I.	1053	Schreiberová L	255
Rašović A.	463		263
	483		333
	563	Sečanski M.	527
Rilak A.	1049	Sedov I.	733
Rimmer P.B.	763		917
Ristić I.	749	Sejdinović D.	665
Ristić P.	881	Senčanski J.	367
Ristović I.	641		375
Rodić A.	459	Serdiuk I.E.	579
Rodić M.V.	881	Severinov K.	455
Rodić O.	455	Shevtsov D.S.	403
Rogov V.A.	201		411
Romanowska A.	141	Shipilova A.S.	63
	939	Shlyapin D.A.	205
Roshal A.D.	579		209
Rožić Lj.	605	Shubin Y.V.	205
Rupar J.	379	Siesler H.W.	73
	383	Simić M.	547
Ruzić D.	101		1019
	923	Simonović B.R.	1007
Salmén L.	531		1011
Salvestrini S.	805	Simonović	
Sandić Z.P.	753	Radosavljević J.	531
Sarap N.	829	Sinadinović-Fišer S.	685
Sarvan M.	153		715
Savić B.	785	Skala D.	193
Savić D.	491	Škapin S.D.	237
Savić T.	229		741
Savić T.D.	677	Slatinska O.	527
Savović J.J.	85	Slavković-Beškoski L.	809

Slavnić D.	467	Starokon E.V.	205
Šljivić-Ivanović M.	789	Stefanović I.S.	745
Šljukić B.	391		753
	399		757
Smarsly B.	159	Stevanić J.	531
Šmelcerović A.	463	Stevanović K.	325
	483		341
Smičiklas I.	789		829
Smirnova N.N.	609	Stoiljković M.	133
Šolević-Knudsen T.	955	Stojadinović S.	237
	959		673
Spasić A.M.	313	Stojanović M.	559
Spasojević D.	801		563
Spasojević J.	495		567
Sredojević D.	153	Stojanović S.	491
Sretenović D.	575		605
Stamenović U.	391	Stojiljković D.	1027
	741	Stojilković K.	931
Stanciu I.	723	Stojković-Simatović	
Stanisavljev D.	321	I.	237
	341		367
	499		407
Stanković B.	689	Stojmenović M.	641
	693	Stoyanovskii V.O.	20
	697		645
Stanković D.	971	Surovikin Yu.V.	209
	975	Suručić Lj.T.	753
	979	Sužnjević D.Ž.	395
Stanković D.M.	793		415
	1045	Syrov E.V.	613
Stanković M.	523	Szabo E.	304
	653	Szakacs Zs.	629
	657		633
Stanković Petrović J.	515		637
Stanojević A.	296	Tabakova T.	197
	317		213
Stanojević Lj.	1027	Tadić N.	693

Talić S.	781	Uskoković D.	237
	853	Uskoković Marković S.	367
Tanasić J.	749		983
Tanasković S.	471	Vasić I.	149
Tanasković S.B.	895	Vasić V.	665
	903		669
Tančić P.I.	673		705
Tarczali-Sarudi P.	217	Vedyagin A.A.	41
Tasić G.	785		201
Temerev V.L.	205		205
	209		645
Tennyson J.	763	Veličković S.	909
Teofilović V.	685		913
	715		1049
Todoran D.	637	Velikić Z.	727
Todoran R.	637	Velikić Z.	499
Todorović M.	833	Velinov N.	593
	881		597
Tomovska R.	585		681
	625	Veljković F.	909
Tošić S.	1031		913
	1035		1049
	1053	Venvik H.J.	241
Tošović J.	125	Veselinović A.M.	555
Tóth A.	312	Veselinović D.	727
Trajanović M.	891		821
Trenikhin M.V.	209	Veselinović L.J.	237
Trifunović N.	421	Vesković A.	451
Trifunović S.R.	93		487
Trifunović S.R.	113		519
Trtica M.	85	Vladimirov S.	379
	133		935
Trtić-Petrović T.	825		951
	859	Vodnik V.	391
	863		705
Tsyulnikov P.G.	209		741
Urban S.	159		

Volodin A.M.	205	Xu Z.	609
	645	Yurchenko S.N.	763
Vorob'eva V.	67	Zadykowicz B.	141
Vraneš M.	863		939
Vranješ-Đurić S.	971	Zadykowicz B.	137
	975	Zarcyn I.D.	403
	979		411
Vrvić M.M.	737	Zarić S.D.	571
Vujačić Nikezić A.	503	Zarić M.	435
	511		535
	705		539
Vujasin R.	475	Zavrazhnov A.Y.	145
	809	Zdolšek T.	859
	813	Zdujčić M.	193
Vujković M.	367	Zeković Z.	1011
	375	Zelenaya A.	67
	407	Žeradjanin A.	963
Vukadinović A.	971	Ziganshin M.A.	947
	975	Zigar D.	4214
	979	Živanović S.C.	233
Vukčević M.	649		491
	817	Živković J.V.	555
Vukelić N.	467	Živković M.D.	149
Vukić N.	715	Živković S.	133
	749	Živković-	
Vukmirović Z.	47	Radovanović V.	903
Vukojević V.	296	Zlatanović S.	109
	1045		1007
Vukosavljević P.	1023		1023
Vulić T.	245	Zlatković M.	1027
Vurdelja B.	913	Zvezdanović J.	1027
Walde P.	3		
Waldmann I.P.	763		
Wera M.	579		
Wester R.	117		
Wilson M.	711		
Wiśniewski A.	279		

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