# MATERIALS and GEOENVIRONMENT

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#### **Historical Review**

More than 90 years have passed since the University Ljubljana in Slovenia was founded in 1919. Technical fields were united in the School of Engineering that included the Geologic and Mining Division, while the Metallurgy Division was established only in 1939. Today, the Departments of Geology, Mining and Geotechnology, Materials and Metallurgy are all part of the Faculty of Natural Sciences and Engineering, University of Ljubljana.

Before World War II, the members of the Mining Section together with the Association of Yugoslav Mining and Metallurgy Engineers began to publish the summaries of their research and studies in their technical periodical Rudarski zbornik (Mining Proceedings). Three volumes of Rudarski zbornik (1937, 1938 and 1939) were published. The War interrupted the publication and it was not until 1952 that the first issue of the new journal Rudarsko-metalurški zbornik - RMZ (Mining and Metallurgy Quarterly) was published by the Division of Mining and Metallurgy, University of Ljubljana. Today, the journal is regularly published quarterly. RMZ - M&G is co-issued and co-financed by the Faculty of Natural Sciences and Engineering Ljubljana, the Institute for Mining, Geotechnology and Environment Ljubljana, and the Velenje Coal Mine. In addition, it is partly funded by the Ministry of Education, Science and Sport of Slovenia.

During the meeting of the Advisory and the Editorial Board on May 22, 1998, Rudarsko-metalurški zbornik was renamed into "RMZ – Materials and Geoenvironment (RMZ – Materiali in Geookolje)" or shortly RMZ – M&G. RMZ – M&G is managed by an advisory and international editorial board and is exchanged with other world-known periodicals. All the papers submitted to the RMZ – M&G undergoes the course of the peer-review process.

RMZ – M&G is the only scientific and professional periodical in Slovenia which has been published in the same form for 60 years. It incorporates the scientific and professional topics on geology, mining, geotechnology, materials and metallurgy. In the year 2013, the Editorial Board decided to modernize the journal's format.

A wide range of topics on geosciences are welcome to be published in the RMZ – Materials and Geoenvironment. Research results in geology, hydrogeology, mining, geotechnology, materials, metallurgy, natural and anthropogenic pollution of environment, biogeochemistry are the proposed fields of work which the journal will handle.

#### Zgodovinski pregled

Že več kot 90 let je minilo od ustanovitve Univerze v Ljubljani leta 1919. Tehnične stroke so se združile v tehniški visoki šoli, ki sta jo sestavljala oddelka za geologijo in rudarstvo, medtem ko je bil oddelek za metalurgijo ustanovljen leta 1939. Danes oddelki za geologijo, rudarstvo in geotehnologijo ter materiale in metalurgijo delujejo v sklopu Naravoslovnotehniške fakultete Univerze v Ljubljani.

Pred 2. svetovno vojno so člani rudarske sekcije skupaj z Združenjem jugoslovanskih inženirjev rudarstva in metalurgije začeli izdajanje povzetkov njihovega raziskovalnega dela v Rudarskem zborniku. Izšli so trije letniki zbornika (1937, 1938 in 1939). Vojna je prekinila izdajanje zbornika vse do leta 1952, ko je izšel prvi letnik nove revije Rudarsko-metalurški zbornik – RMZ v izdaji odsekov za rudarstvo in metalurgijo Univerze v Ljubljani. Danes revija izhaja štirikrat letno. RMZ – M&G izdajajo in financirajo Naravoslovnotehniška fakulteta v Ljubljani, Inštitut za rudarstvo, geotehnologijo in okolje ter Premogovnik Velenje. Prav tako izdajo revije financira Ministrstvo za izobraževanje, znanost in šport.

Na seji izdajateljskega sveta in uredniškega odbora je bilo 22. maja 1998 sklenjeno, da se Rudarsko-metalurški zbornik preimenuje v RMZ – Materiali in geookolje (RMZ – Materials and Geoenvironment) ali skrajšano RMZ – M&G. Revijo RMZ – MG upravljata izdajateljski svet in mednarodni uredniški odbor. Revija je v vključena v mednarodno izmenjavo svetovno znanih publikacij Vsi članke so podvrženi recenzijskemu postopku.

RMZ – M&G je edina strokovno-znanstvena revija v Sloveniji, ki izhaja v nespremenjeni obliki že 60 let. Združuje področja geologije, rudarstva, geotehnologije, materialov in metalurgije. Uredniški odbor je leta 2013 sklenil, da posodobi obliko revije.

Za objavo v reviji RMZ – Materiali in geookolje so dobrodošli tudi prispevki s širokega področja geoznanosti, kot so: geologija, hidrologija, rudarstvo, geotehnologija, materiali, metalurgija, onesnaževanje okolja in biokemija.

Glavni urednik

Editor-in-Chief

### FSW of aluminium alloy AlSi12

Varjenje s trenjem in mešanjem aluminijeve zlitine AlSi12

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#### Abstract

FSW of 4 mm thick casting aluminium alloy AlSi12 and dissimilar welding of AlSi12 with Al 99.5 in butt joint was done. A classical tool was used with tilt angle of 2°, tool rotation speed varied from 235 r/min to 1 180 r/min, welding speed between 71 mm/min to 450 mm/min and joint gap width from 0 mm to 0.5 mm. From the produced welds, samples for microstructure analysis were prepared for observation on a light microscope under the polarized light source. A Vickers micro-hardens was measured across the weld. Analysis of microstructure, hardness measurement and tensile test of FSW welds were done. A set of FSW welding parameters was determined at which acceptable welds were obtained. Dissimilar welding of AlSi12 with Al 99.5 is possible.

**Key words:** friction stir welding, aluminium alloy EN AW 4430A (AlSi12), microstructure, mechanical properties, dissimilar welding

#### Izvleček

V prekrovnem spoju smo s trenjem in mešanjem varili 4 mm debelo pločevino iz aluminijeve zlitine AlSi12 ter AlSi12 z Al 99,5. Uporabili smo klasično orodje s čepom. Spreminjali smo hitrost varjenja orodja med 235 r/min in 1 180 r/min, hitrost varjenja pa je bila med 71 mm/min in 450 mm/min, nagibni kot orodja 2°, širina špranje pa 0 mm in 0,5 mm. Iz zvarov smo izdelali vzorce za analizo mikrostrukture ter merjenje natezne trdnosti. Mikrostrukturo vzorcev smo pregledali z optičnim mikroskopom v polarizirani svetlobi in izmerili trdoto po Vickersu. S parametrično analizo smo določili procesno okno varilnih parametrov, da lahko dobimo kakovostne zvarne spoje. Spajanje aluminijeve zlitine AlSi12 s čistim aluminijem Al 99,5 je mogoče.

**Ključne besede:** varjenje s trenjem in mešanjem, aluminijeva zlitina EN AW 4430A (AlSi12), mikrostruktura, mehanske lastnosti, varjenje različnih materialov

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#### Introduction

One of the current trends and goals of transportation industry is to lower the green gas emissions, increase fuel economy and use renewable energy. This is partly achievable by a) reduction of car body weight, b) engine volume and weight downsizing, c) the use of hybrid engines and/or d) electric engines. Weight reduction could be achieved using materials with favorable weight to strength ratio (Al alloys, Mg allovs, high strength steels etc.). Especially in a production of hybrid engines there is a demand for heat exchangers, cooling units or other closed shapes products for cooling of electric components and other various purposes. These products have complex shape and needs to be light, produced in high series, with sharp dimensional tolerances, and with low production costs. This is easily done using high pressure die casting technology (HPDC). With the HPDC closed parts cannot be produced directly, but some sort of joining must be used. Screw fastening is time consuming and expensive. Adhesive bonding has limitations in strength at higher temperature. Arc welding (MIG) of castings is demanding due to lubrication of the molds during HPDC i.e. castings, which produces metal explosions during arc welding and pores in the weld, which can cause leaking. TIG welding is slightly easier, producing less porosity in the weld.

FSW is a promising technology for joining castings made of different casting alloys. The advantages of FSW are that a) a sound welds can be made (without pores), b) no material explosions are present due to absence of molten metal and c) no filler material and shielding gasses are needed. The disadvantage is the pricier weld due to higher investment cost, tools and clamping devices. A weldability of different casted aluminium alloys is studied, regarding microstructure and mechanical properties<sup>[1-11]</sup>. Results showed that welding is possible, but there are the limitations regarding welding parameters. Optimization of welding parameters using Taguchi methods showed that the tool rotation speed has the highest influence of the strength of the weld and the second influencing parameter is welding speed<sup>[12]</sup>. FSW welding of dissimilar aluminium alloys is more demanding and has a smaller processing window.

The aim of the research was to analyze weldability of AiSi12 in die casted condition. Aluminium alloy AlSi12 has a good castability, machinability and strength and limited weldability using arc welding technique. It is resistant to chemically aggressive liquids, has a low melting temperature, and is good for producing thin-walled castings of complex geometry. FSW welding parameters i.e. tool rotation speed, welding speed and tilt angle have the influence on the formation of welding defects, weld apices appearance, microstructure and weld strength. The aim of this research is to discover the welding parameters providing weld microstructure without defects. FSW was done at tool rotation speed from 235-1180 r/min, welding speed from 71-450 mm/min and the tool tilt angle was held constant at 2°. Factors feed per revolution (FPR) and revolutions per feed (RPF) were introduced to get the better insight into the friction stirring process. The RPF gives the information about heat input per weld length. Miniature samples for tensile testing were prepared from the welds. Welds were examined under the light optic microscope and Vickers hardness was measured.

#### **Materials and methods**

# Dimensions and chemical composition of workpieces

The standard EN AW 4430A (AlSi12) aluminium alloy with chemical composition in mass fractions according to the standard (0.55% Mn, 10.5-13.5% Si, 0.08% Cu, 0.45-0.9% Fe, 0.15% Zn, 0.15% Ti and the rest Al) in die casted state was used for testing. The workpiece dimensions were 160 mm × 55 mm × 4 mm. Physical and mechanical properties of the alloy were not determined but taken according to standard (Table 1)<sup>[13]</sup>.

#### FSW tool

The FSW tool was made from standard EN 42CrMo4 steel<sup>[14]</sup>. A basic FSW tool geometry was used with treaded pin 3.9 mm long (M6  $\times$  1.5) and the concave shoulder

**Table 1:** Physical and mechanical properties of HPDC alloy AlSi12<sup>[13]</sup>

Label	ρ (kg/m³)	<i>R</i> <sub>m</sub> (МРа)	<i>R</i> <sub>р0.2</sub> (МРа)	λ (W/m K)	T <sub>sol</sub> (°C)	$T_{\text{casting}}$ (°C)
AlSi12	2 680	170-240	80-130	130-160	570-580	650-700

( $\Phi$  = 16 mm) for producing pressure under the tool shoulder (Figure 1). After machining the tool was surface quenched and tempered to get the surface hardness of 58 HRc.

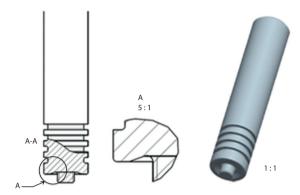


Figure 1: FSW tool geometry.

#### Friction stir welding

A plan of experiments was prepared regarding capabilities of universal milling machine used (Prvomajska ALG 200). Different combinations of tool rotations and welding speeds were tested at constant tilt angle of 2°. The FSW tool rotated from 235 r/min to 1 180 r/min, and the welding speeds changed from 71 mm/min to 450 mm/min. Factor of feed per revolution (FPR) in mm/r and revolution per feed (RPF) in r/mm were introduced for better distinguishing between different welding parameters. The FPR varied from 60 mm/r to 1 620 mm/r. RPF, which represents the "frictional heat input" per weld length, was between 16.16-0.62 r/mm. A backing plate underneath the workpiece enabled the formation of pressure under the tool shoulder by preventing the aluminium alloy to flow away from the seam. The two workpices were clamped in a vice.

#### Preparation of samples and welding

From the FSW welds a miniature tensile test samples were sectioned perpendicular to the welding direction, and a weld cross-sections for analysis of microstructure and macrtostructure. Before sectioning the samples with water jet, the workpiece surfaces were milled to remove weld underfill and toe flash.

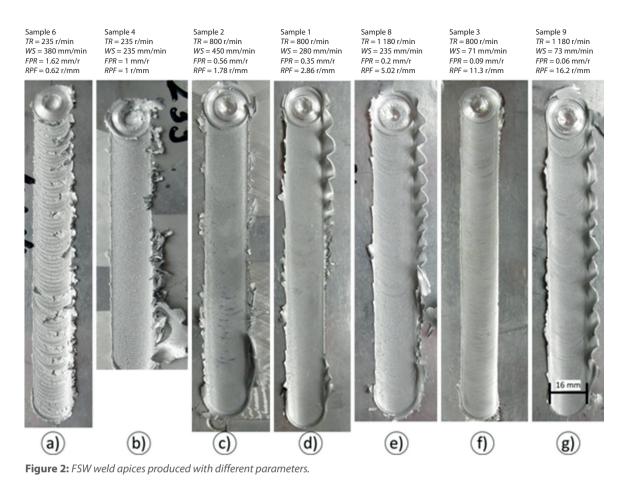
The uniaxial tensile tests were done using computer controlled Zwick/Roell Z050 tensile testing machine. Measurements were done using Testexpert software. The strain was measured with extensometer directly on the sample.

The samples for analysis of microstructure and microstructure were sectioned, grinded and polished. The samples for macrostructure analysis were etched using Keller reagent (1125 ml HCl, 558 ml HNO<sub>3</sub>, 200 ml HF and 1500 ml H<sub>2</sub>O) and the microstructure was analysed using a light optic microscope. Samples for microstructure analysis were anodizing with Baker's reagent. The microstructure was examined using a light optic microscope under polarised light and with the digital camera for picture acquiring. A Vickers micro-hardness HV1 (load is 9.807 N) was measured across the welds.

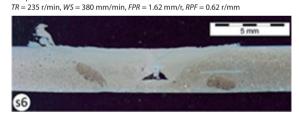
#### **Results and discussion**

# Dimensions and chemical composition of workpieces

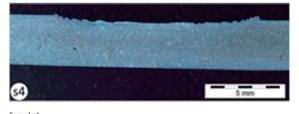
Figure 2 shows the FSW weld apices. The end of the weld is indicated with a hole, which is a negative of the FSW tool pin. Visual assessment of the weld apices reveals smooth weld apices for FPR between 60 mm/r and 560 mm/r i.e. for RPF between 16.2 r/min and 1 r/mm (Figure 3). At sample 9 (Figure 2g) frictional heat input was the highest (RPF = 16.2 r/mm). At this sample a tool moved a bit too much into the workpiece, due to higher frictional heat input, which softened the material. At samples 3, 8, 1 and 2 (Figures 2f, e, d, and c) the RPF was between 11.3 r/mm and 1.78 r/mm and weld apices were smooth. When the tool speed increased to a 1 r/mm and 0.44 r/mm (Figures 2b, a) the heat input become smaller and weld apices become rough with the traces of material tearing.



Sample 6



Sample 4 TR = 235 r/min, WS = 235 mm/min, FPR = 1 mm/r, RPF = 1 r/mm



Sample 1 *TR* = 800 r/min, *WS* = 280 mm/min, *FPR* = 0.35 mm/r, *RPF* = 2.86 r/mm



Figure 3: FSW weld macrostructure presented according to the heat input.

Sample 8 TR = 1 180 r/min, WS = 235 mm/min, FPR = 0.2 mm/r, RPF = 5.02 r/mm



Sample 3

TR = 800 r/min, WS = 71 mm/min, FPR = 0.09 mm/r, RPF = 11.3 r/mm



Sample 9 *TR* = 1 180 r/min, *WS* = 73 mm/min, *FPR* = 0.06 mm/r, *RPF* = 16.2 r/mm

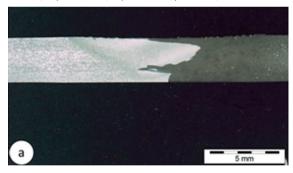


#### Weld microstructure

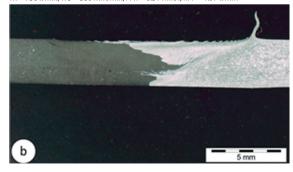
Macrographs of the selected FSW welds are shown on the Figure 3. The die casted base alloy had plenty of pores, which vanished in the weld. Figure 3 s6 presents the macrograph of sample 6, welded with the smallest frictional heat input (*RPF* = 0.62 r/mm). The "worm hole" i.e. "tunnelling" defect appears if welding is done with insufficient heat input or if welding force in the axial direction is not big enough. If heat input was higher than 1 rev/min no such defect was present. At samples s1 and s8 (Figure 3) a small cavity is present closer to the weld apices. The probable reason for that is insufficient tool penetration i.e. pressure under the tool shoulder or force in z-direction. Samples s4, s3 and s9 (Figure 3) represents quality weld without defect, except of small under-fill and toe flash, as a result of higher heat input.

Figure 4 shows a macrostructure of two dissimilar welds obtained at a) *RPF* 1.25 r/mm with AlSi12 on the advancing side, and b) *RPF* = 1.97 r/mm with Al 99.5 on the advancing side. In both cases a quality weld, with good mixing





Sample 13 TR = 750 r/min, WS = 380 mm/min, FPR = 0.51 mm/r, RPF = 1.97 r/mm



**Figure 4:** *Dissimilar FSW weld between AlSi12 and Al 99.5: a) AS* = *AlSi12, b) AS* = *Al 99.5.* 

of both materials is obtained. The weld shows finer microstructure compared to either of the two base metals, and less porosity. Weld on Figure 4a was produced with less heat input and has no under-fill is present. At weld (Figure 4b) produced at higher heat input a toe flash is present at the advancing side of the weld due to higher heat input and better forming ability of the pure aluminum.

Figure 5 shows weld microstructure in the weld, HAZ and base alloy. The grain size at weld apices (Figures 5b, c) is very small due to vicinity of tool shoulder i.e. heat generation. Small sized grains are obtained across the whole weld (Figure 5a). At weld root material is not stirred to the bottom of the workpiece (Figure 5d). The oxide surface of contacting workpices is clearly seen. Such oxide line/layer could present initiation site for cracking during loading or exploitation.

#### Hardness

The Vickers hardness HV1 was measured across the weld at the middle of the weld (2 mm below the surface) in the total distance of 26 mm. The hardness is shown for the samples 0, 7, 1 and 6 (Figure 6). The centre of the weld is shown with the "dash-dot" line and the advancing side of the weld is on the right side of the plot of the Figure 6. When welding with higher frictional heat input of 16.2 r/mm, the whole workpiece was heated above the temperature of recrystallization, where a grain growth occurs. The hardness was the lowest among all compared samples ( $\approx 80$  HV1 in the base metal and HAZ, and 60 HV1 in the weld). When welding with optimal welding parameters (sample 7 and 1), the hardness across the weld was slightly higher (63–73 HV1) and was smaller than in the base alloy. A higher frictional heat input at the advancing side of the weld is resulting in a slightly lower hardness in the HAZ ( $\approx$  75 HV1). When frictional heat input was very low ( $\approx 0.62 \text{ r/mm}$ ) weld hardness was 68 HV1.

At dissimilar weld (Figure 6 s13) with Al 99.5 on the advancing side hardness is decreasing to the softer Al 99.5, where hardness of 60 HV is obtained in the base metal.

#### Tensile properties

The tensile strength of the base alloy used for experimental workpiece was not measured, but taken from the literature data (Table 1). Yield strength of AlSi12 aluminium alloy is between 80 MPa and 130 MPa and ultimate tensile strength between 170 MPa and 240 MPa (Table 1). Since a non-standard test specimens were used, the results could hardly be compared with the results from the literature. The ultimate tensile strength of tensile test specimens was generally in the range of the base aluminium alloy. When welding with high heat input more than 11.3 r/mm (sample 3 and 9), the tensile strength was slightly higher than the lowest standard value i.e. 180 MPa. If welds were without defects, a higher tensile strain was obtained.

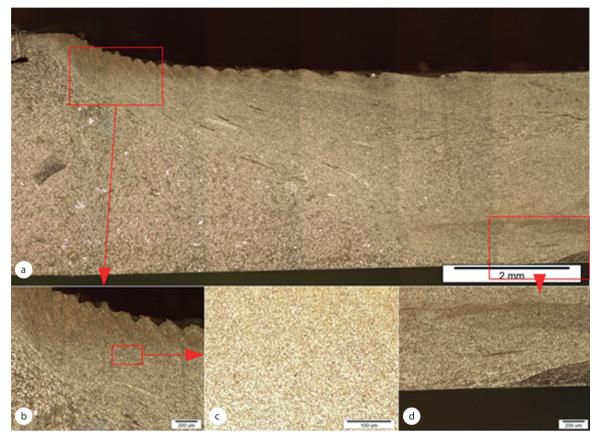
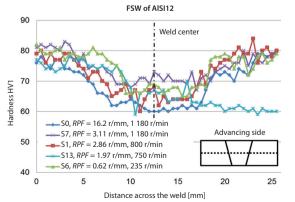


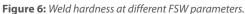
Figure 5: Microstructure (polarised light microscopy images) of FSW weld produced at 1 180 r/min RPF = 3.11 r/mm (sample 7): a) weld with HAZ and base alloy, b) weld apices and HAZ, c) weld and d) weld root.

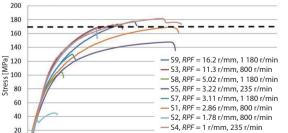
20

0

0







5

4

Strain [%]

Tensile test - perpendicular to the direction of welding

Figure 7: Results of tensile test.

2

3

1

S6, RPF = 0.62 r/mm, 235 r/min

8

Based on the analysed results the following can be summarized:

The tensile strength of the weld and base metal was similar in most cases. If the weld had a cavity, a low tensile strength was measured. Lower tensile strength than reference value was measured due to the porosity of base alloy, in which case the test sample broke in the base alloy and not in the weld. Tensile strength was in the range of base alloy, if the welds had no cavities, pores or other defects.

If RPF < 1 r/mm, an elongated cavity i.e. "worm hole" defect is present due to too low heat input. Quality welds (without cavities) are obtained if RPF > 1 r/mm (FPR < 1 mm/r). If RPFis high (> 5 r/mm) a toe flash and under-fill is possible due to high frictional heat input.

FSW welds had a finer microstructure compared to the base alloy, and were without pores even if the base alloy had them.

Welds were softer (60–70 HV1) compared to base alloy (80 HV1).

The friction coefficient changes with tool rotation speed. A higher heat input was noted at higher rpm at the same *RPF/FPR* rates.

#### Acknowledgements

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### **Production of Ni-Mn-Ga foam**

Izdelava porozne zlitine Ni-Mn-Ga

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#### Abstract

Ni-Mn-Ga alloys presents the most promising materials among the functional metallic materials showing the magnetic field induced strain (MFIS) especially due to recent discovery of the effect of porosity in this material. This work presents the production of the Ni-Mn-Ga alloy foam via casting replication method using the sodium aluminate as a space holder for the later porosity and their characterization. Two different compositions of Ni-Mn-Ga alloys and space holders were produced. Space holders were prepared in one case of powder size rage of 500  $\mu$ m to 600  $\mu$ m and in the other case of 63  $\mu$ m to 100  $\mu$ m and 315  $\mu$ m to 500  $\mu$ m powder sizes. Samples microstructure was characterized after the alloy infiltration in to a sintered space holder to determine alloy and sodium aluminate fraction.

**Key words:** magnetic shape memory, casting, Ni-Mn--Ga- alloy, porous alloy

#### Izvleček

Zlitine iz sistema Ni-Mn-Ga spadajo med bolj obetavne materiale iz skupine funkcionalnih kovinskih materialov. Ti materiali kažejo pojav spremembe dimenzij, induciranih z magnetnim poljem, ki je zanimiv predvsem v povezavi z nedavno odkritim vplivom poroznosti. V delu je predstavljen postopek izdelave zlitine Ni-Mn-Ga in infiltracije zlitine v porozno predformo iz natrijevega aluminata ter karakterizacija. Izdelali smo dve različni zlitini in predformi. Predforma je bila v enem primeru izdelana iz delcev velikosti med 500  $\mu$ m in 600  $\mu$ m, v drugem pa med 63  $\mu$ m in 100  $\mu$ m ter 315  $\mu$ m in 500  $\mu$ m. Po infiltraciji zlitine v porozno predformo natrijevega aluminata je potekala karakterizacija mikrostrukture, pri čemer sta bila določena deleža zlitine in natrijevega aluminata.

**Ključne besede:** magnetni oblikovni spomin, ulitek, zlitina Ni-Mn-Ga, porozna zlitina

#### Introduction

An important group of functional metallic materials are shape memory alloys. There are several ferromagnetic alloys which are capable of so called magnetic-field-induced strain (MFIS) like Dy, Fe-Pd alloy, Fe<sub>2</sub>Pt and Ni-Mn-Ga alloys.<sup>[1]</sup> These materials are capable of changing the shape or dimensions under the externally applied magnetic field.<sup>[1, 2]</sup> The most promising material among these are Ni-Mn-Ga alloys. Monocrystalline Ni-Mn-Ga alloys show very large MFIS up to a 10 %. MFIS effect (strain) in these alloys is produced by moving of twin boundaries caused by internal stresses which are generated by magnetic anisotropy energy.<sup>[3]</sup> These twin boundaries in this alloy form after the diffusionless martensitic type phase transformation from L2, ordered type structure.<sup>[4]</sup> Fine grained non-textured polycrystalline alloys, on the other hand, shows nearly zero MFIS effect.<sup>[1]</sup> Nearly zero MFIS effect in fine grain polycrystalline alloy is consequence of the fact that grain boundaries effectively suppress the motion of twin boundaries and that neighbouring grains in non-textured alloy have incompatible MFIS.<sup>[1]</sup> MFIS in these alloys can be increased by increasing the grain size and introducing the texture. Recently it was also found and explained that large MFIS (2-9 %) in these polycrystalline alloys can be gained by the introduction of porosity smaller than the grain size.<sup>[2, 3, 5, 6]</sup>

Main aim of this paper is to present the synthesis of the Ni-Mn-Ga alloy foam via casting replication method<sup>[7]</sup> using the sodium aluminate as a space holder for the later porosity and their characterization.

#### Materials and methods

The Ni-Mn-Ga alloy foams were prepared by infiltration of liquid alloy in to a sintered sodium aluminate (NaAlO<sub>2</sub>) space-holder. Sodium aluminate powders of the specific sizes were prepared from the commercial sodium aluminate ( $Al_2O_3 \cdot Na_2O$  or  $NaAlO_2$ ) powder (purchased from Alfa Aesar). Commercial powder was first sintered at 1 550 °C for 3 h in air to produce the bulk material. Bulk material was milled in vibrating mill for short time (couple of seconds) to produce the powder of appropriate size distribution. Powder was later sieved to extract the powders with sizes ranging between 63  $\mu$ m to 100  $\mu$ m, 100  $\mu$ m to 200  $\mu$ m, 315  $\mu$ m to 500  $\mu$ m, and 500  $\mu$ m to 600  $\mu$ m.

Space holder composed of two different sizes of the particles was prepared by dry mixing of powders with sizes of 63  $\mu$ m to 100  $\mu$ m and 315  $\mu$ m to 500  $\mu$ m. Mixed powders were poured in to an alumina crucible with diameter of 11 mm to height of approximately 5 mm and sintered at 1 550 °C for 3 h in air. The space holder specimen produced of monosized distribution powder was prepared by pouring the powder of 500  $\mu$ m to 600  $\mu$ m in to an alumina crucible with diameter of 11 mm to height approximately 5 mm. Space holder was in this case sintered at 1 580 °C for 3 h.

Ni-Mn-Ga alloy was synthesized from pure nickel (mass fractions w = 99.83 %), manganese (99.9 %) and gallium (99.999 %) using arc melting furnace (Compact Arc Melter MAM-1, Edmund Bühler GmbH). Pieces of the arc melted alloy were put on the top of the space holder within the alumina crucible and heated in a vacuum to a temperature of 1 250 °C and held at the temperature for about 30 min. Afterwards the Ar was introduced in to a furnace chamber to gain the pressure of 1 bar which forced the alloy in to a porous space holder. After the infiltration the furnace was cooled down to an ambient temperature.

Infiltrated samples were cold-mounted, ground and polished. Metallographic observation has been performed using light microscopy for the evaluation of the distribution of the sodium aluminate (porosity) and determination of the fraction of the sodium aluminate (porosity). The presence of twinning which is characteristic feature of the non-cubic phase at the ambient temperature was represented by polarized light microscopy. Light microscope ZEISS AXIO Imager.A1m combined with software for digital image processing Axio Vision was used in this work. Scanning electron microscope Jeol JSM 5610 equipped with energy dispersion spectrometer (EDS) Gresham scientific instrument model no.: Sirius 10/SUTW was used for determining compositions of synthesized alloys.

#### **Results and discussion**

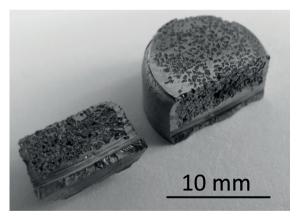
Beside samples presented in this work more sintered space holder samples were prepared. For the successful infiltration the space holder must be adequately sintered, which means that particles in space holder are attached to each other and that there is still enough open porosity between them which enables successful infiltration. Based on previously mentioned samples it was found that appropriate conditions for sintering of space holder are between 1 550 °C and 1 580 °C for 3 h. Higher temperature limit stands for the space holder composed only of large particles while lower temperature stand for the space holder composed of large and small particles. This finding is also in the agreement with the fact that sintering temperature also depends on the particles size which means that smaller particles lead to a lower temperature of sintering.

Typical microstructures of the sample T23 prepared of the preform composed of 500  $\mu$ m to 600  $\mu$ m particles are presented in Figure 1. Microstructures in Figure 1 show that sodium aluminate particles are rather evenly distributed with slightly different fraction in different regions of the sample. This was also confirmed by the determination of the fraction of the alloy presented in Table 1. As presented in Table 1 the average fraction of the alloy and sodium aluminate are 38 % and 62 % in sample T23.

**Table 1:** Fraction of alloy and sodium aluminate in the sample T23 prepared of the preform composed of 500 μm to 600 μm particles

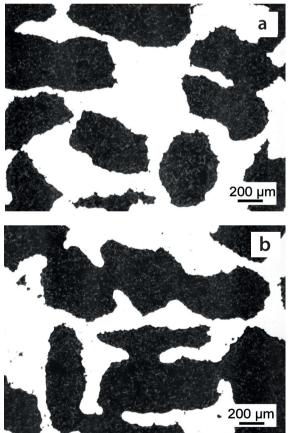
	Fraction (	%)
	Sodium aluminate	Alloy
M1	58.7	41.3
M2	65.2	34.8
Average	62.0	38.0

Macrostructure of the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63  $\mu$ m to 100  $\mu$ m and 315  $\mu$ m to 500  $\mu$ m is presented in Figure 2. Macrostructure in this figure shows that the central region of the sample was not completely infiltrated.



**Figure 2:** Macrostructure of the sample T3 prepared of the preform composed of the particles with sizes ranging between 63 μm to 100 μm and 315 μm to 500 μm.

Microstructures of the sample T3 are presented in Figure 3. It can be seen from the microstructures that the distribution of small sized particles is not even. Quantity of small sized particles is also too small to enclose the large



**Figure 1:** Microstructures of sample T23 prepared of the preform composed of 500 µm to 600 µm particles with a) higher and b) lower fraction of the alloy.

particles and it should be increased in further work. On the other hand, large particles are more evenly distributed. The exception is the central region of the sample where alloy did not completely filled the sample and we cannot judge, based on these figures, whether dark regions are only large particles or there are large particles with unfilled space. Locally denser space holder can lead to a locally lower fraction of alloy or it can even prevent further progression of alloy in to a preform. If progression of the alloy is the reason, it could be overcome by higher pressure of infiltration or smaller size of the sample. Uneven distribution of small sized particles is probably also the consequence of the way the powders were mixed (dry mixing) and poured in to an alumina crucible. Zhang et al. report that even distribution of the particles can be accomplished by the introduction of powders to crucible layer by layer in the acetone.<sup>[5]</sup>

200 µm'

b 200 µm

Figure 3: Microstructures of the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63 µm to 100 µm and 315 µm to 500 µm. a) represents periphery of the sample and b) centre of the sample.

Fraction of the alloy and the sodium aluminate in the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63 µm to 100 µm and 315 µm to 500 µm is presented in Table 2. Results show that there is a difference in alloy fraction between the central and periphery regions of the sample. As it is given in the Table 2 the average fraction of the alloy and sodium aluminate in this sample are 31.4 % and 68.6 %. Comparison of these results to results for sample T23 prepared of monosized space holder shows that in the case of monosized space holder fraction of the alloy is larger. This result is expected since the powders of several different sizes which are properly mixed in comparison to monosized possess higher green density.

Table 2: Fraction of the alloy and the sodium aluminate in the sample T3 prepared of the space holder composed of the particles with sizes ranging between 63 µm to 100 µm and 315 um to 500 um

	Fraction (%	6)
	Sodium aluminate	Alloy
M1	64.4	35.6
M2	66.3	33.7
M3	70.1	29.9
M4	73.7	26.3
Average	68.6	31.4

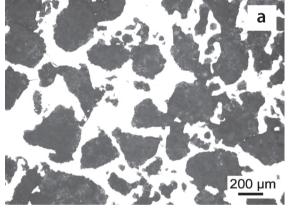
Different compositions of alloys were used in these experiments. Compositions of two alloys determined by EDS after remelting under controlled conditions (10 K/min in an atmosphere of Ar gas) are presented in Table 3 and their microstructures are presented in Figure 4.

**Table 3:** Compositions of the alloys A and B determined by
 EDS in mole fractions (x/%)

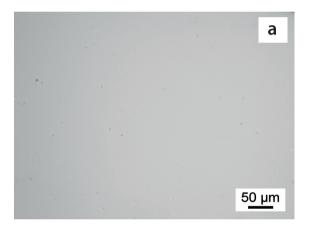
	Alloy A	Alloy B
Ni	49.7	56.0
Mn	26.9	23.2
Ga	23.4	20.8

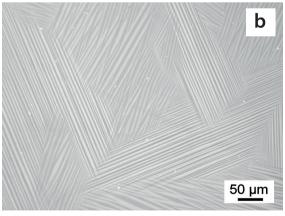
Ni-Mn-Ga alloys undergo diffusionless martensitic type of phase transformation which is necessary for the MFIS effect. Martensite start temperature in these alloys depends on the composition of the alloy.<sup>[4, 8]</sup> It was assumed that the stability of  $\beta$ -Ni<sub>2</sub>MnGa is controlled by





Hume-Rothery electron compound mechanism and it was shown that linear relationship exists between martensite start temperature Ms and electron/atom (e/a) concentration.<sup>[4, 8]</sup> Using such a relationship presented by Schlagel et al.<sup>[8]</sup> and compositions of alloys determined by EDS give us (e/a) concentration in the case of the alloy A of 7.56 and the martensite start temperature in the rage between 200 K and 250 K. This temperature is below the ambient temperature. In the case of the alloy B (e/a) concentration and the martensite start temperature are 7.85 and approximately 450 K. Polarised light microscopy images presented in Figure 4 shows that twinning, which is characteristic feature of non-cubic martensitic phase, is observed only in the case of the alloy B (Figure 4b). This result is consistent with the previously accessed martensite start temperatures.





**Figure 4:** Polarized light microscopy images of a) alloy A and b) alloy B.

#### Conclusions

It was found, based on the prepared sodium aluminate space holders that the appropriate conditions for sintering of space holder are between 1 550 °C and 1 580 °C for 3 h. High temperature limit was found to stand more for the space holder composed of large particles while lower temperature stand for the space holder composed of large and small particles.

Two samples were prepared by infiltration of the alloy in to a space holder composed of only large particles (500  $\mu$ m to 600  $\mu$ m) and in to a space holder composed of large (315  $\mu$ m to 500  $\mu$ m) and small (63  $\mu$ m to 100  $\mu$ m) particles. Microstructural analysis of the sample composed of only large particles shows rather evenly distributed particles of sodium aluminate with slightly different fraction in different regions of the sample.

Distribution of small sized particles in sample composed of large and small particles is not even and the quantity of small sized particles is also too small. Large particles are more evenly distributed. The exception is the central region of the sample where the alloy did not completely filled the sample. It was found that the average fraction of alloy and sodium aluminate in sample composed only of large particles are 62 % and 38 % while in the case of sample composed of large and small particles are 68.6 % and 31.4 %.

Microstructures of both alloys are consistent with expected martensite start temperatures based on compositions.

#### Acknowledgments

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### Analysis of surface roughness in the Sveta Magdalena paleo-landslide in the Rebrnice area

### Analiza hrapavosti površja fosilnega plazu Sveta Magdalena na območju Rebrnic

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#### Abstract

In the geomorphologic analysis of the Rebrnice area, SW Slovenia, we used a morphometric indicator of surface roughness, which proved to be very useful in the study of the Sveta Magdalena paleo-landslide in the Rebrnice area. For the investigation of the surface roughness on the Sveta Magdalena paleo-landslide and its nearby area, we used two methods in GIS: slope variability and the Terrain Ruggedness Index (TRI). As an input for the analysis of roughness, we used digital elevation models (DEMs) with a resolution of 3 m × 3 m (resampled from 1 m × 1 m cells) obtained by airborne laser scanning.

Based on the analysis of the surface roughness we identified typical morphological characteristics that may reflect the mass gravity flow deposition. With the proper visualization (symbology) we can recognize lobate and fan-shaped forms of the bodies, increased roughness at the edges and at the forefront of the sedimentary body, marginal levees at the edges and arcuate levees in the middle part of the sedimentary bodies.

**Key words:** surface roughness, lidar, GIS, paleo-landslide, debris flow deposit

#### Izvleček

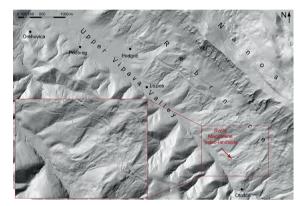
V okviru morfometričnih analiz smo na območju Rebrnic uporabili morfometrični indikator hrapavosti površja, ki se je izkazal za zelo uporabnega pri preučevanju fosilnega plazu Sveta Magdalena na območju Rebrnic. Za preiskovanje površinske hrapavosti na fosilnem plazu Sveta Magdalena in njegovi neposredni okolici smo v GIS-u uporabili dve metodi: metodo variabilnosti naklonov (*slope variability*) in metodo indeksa TRI (*Terrain Ruggedness Index*). Kot podlago za analizo hrapavosti smo uporabili digitalni model višin (DMV) ločljivosti 3 m × 3 m (osnovna ločljivost je bila 1 m × 1 m), pridobljen z zračnim laserskim lidarskim skeniranjem.

Na podlagi analize hrapavosti površja smo prepoznali značilne morfološke karakteristike, ki jih lahko vsebujejo sedimenti drobirskega toka. S primerno vizualizacijo smo lahko prepoznali jezičasto in pahljačasto obliko telesa, povečano hrapavost ob robovih sedimentnega telesa, povečano hrapavost na čelu pahljače, ki lahko pomeni inverzno gradacijo v čelu drobirskega toka, obrobne grebene v pahljači ter usločenost grebenov v sredini sedimentnega telesa.

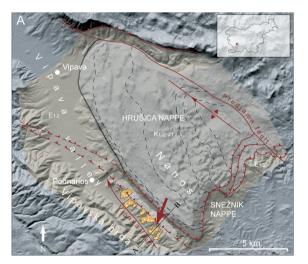
**Ključne besede:** hrapavost površja, lidar, GIS, fosilni plaz, sediment drobirskega toka

#### Introduction

Morphometric analyses and their visualization can be of great help in the characterization of the variability of the surface and are an excellent complement to traditional fieldwork techniques and geological mapping. Morphometry, which is defined as a quantitative measurement of landforms and provides an objective comparison of different segments of the Earth's surface is often used in identifying and defining the Earth's surface, modelling the surface processes and tectonic geomorphology.<sup>[1–3]</sup> Within morphometric analysis, in the Rebrnice area, or more precisely on one of the five sedimentary bodies – Sveta Magdalena paleo-landslide (Figure 1) – we used a morphometric indicator,



**Figure 1:** Digital elevation model of large fan- and tongueshaped sedimentary bodies in the Rebrnice area depicting the study area – Sveta Magdalena paleo-landslide. The image was generated from Lidar data rasterized to a resolution of  $1 \text{ m} \times 1 \text{ m}$ .

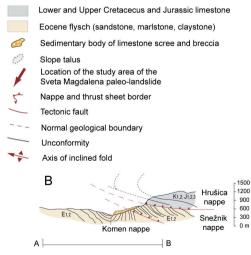


that is, surface roughness, which is, besides the curvature analysis of the surface, very useful in studying and identifying paleo-landslides.<sup>[4-7]</sup> With the analysis of the surface roughness we wanted to distinguish the morphology of the sedimentary body from the surrounding morphology and accurately characterize the surface of sedimentary bodies.<sup>[8-10]</sup>

The article deals with the variability of the quantification of surface roughness, based on two methods: Slope variability<sup>[1]</sup> and the Terrain ruggedness index (*TRI*).<sup>[11]</sup> As the basis for the analysis of surface roughness, we used digital elevation models (DEMs) with resolution of 3 m × 3 m obtained by airborne laser scanning (Figure 1).<sup>[12, 13]</sup> With visual interpretation and field validation, we achieved greater consistency in determining forms of relief structures and their metric properties.

### **Geologic setting**

The research area belongs to a complex Eocene to Oligocene fold-and-thrust structure of the External Dinarides<sup>[14-15]</sup>, with three nappes in the Vipava Valley region (Figure 2): Komen, Snežnik and Hrušica (listed from the structurally lowest to the highest nappes). The Komen and Snežnik nappes are composed solely of flysch, lying in the central part of the valley and in the Vipavska Brda (SW slopes). Mesozoic carbonates of Hrušica nappe are overthrusted on



**Figure 2:** A simplified geological map and cross section across the Upper Vipava valley; from Vipavska Brda to Rebrnice and Nanos; SW Slovenia.<sup>[14, 17]</sup>

Snežnik nappe, represented by Tertiary flysch in the Rebrnice (NE) slopes of the Vipava valley. Geomorphologically, this thrust contact is reflected by steep carbonate cliffs in the upper parts of the slopes, while the middle and lower parts of the Rebrnice area, composed of flysch rocks, are gently sloping. The latter areas are covered by numerous fan-shaped Quaternary deposits<sup>[8, 16]</sup>, some of which were deposited by gravity-flows.<sup>[8, 10]</sup> A large Neogene dextral strike-slip fault zone (up to 300 m wide) known as the Vipava fault is also present in the central part of the valley (Figure 1).<sup>[17]</sup>

#### **Methods and materials**

As a basis for the analysis of surface roughness, we used digital elevation models (DEMs) obtained with airborne laser scanning (ALS). A one-metre resolution DEM was obtained by a combination of adaptive triangulated irregular network densification –  $\text{ATIN}^{[17]}$ ; implemented in Terrasolid Terrascan 11 – and repetitive interpolation – REIN.<sup>[11, 19, 20]</sup> Lidar data with a resolution of a 1 m × 1 m cell were smoothed by the Focal Statistics Spatial Analyst tools, the size of the area of 3 m × 3 m cell.<sup>[13, 20]</sup> From the abovementioned digital elevation model we made out two information layers, namely the slope roughness variation method and the *TRI* method.<sup>[1, 11]</sup>

The slope variability method (here slightly modified to analyze the height instead of slope values, to emphasise the difference in relief) analyses the differences between the lowest and highest elevations in the selected area. By the Focal Statistics tool in ArcGIS, we made a map of these elevations, based on the digital elevation model (DEM) with resolution 3 m × 3 m (resampled from 1 m × 1 m). The final map (Figure 3B) shows the differences between the lowest and highest elevations in the area.

 $SV = S_{\max} - S_{\min}$ 

SV = Slope Variability output raster  $S_{max}$  = maximum height value raster  $S_{min}$  = minimum height value raster The *TRI* method is based on the calculation of relief ruggedness, calculated from differences between the elevations in the cells in the window size of 3 m × 3 m. By the Focal Statistics tool, we produced the raster map of maximum  $(H_{\text{max}})$  and minimum elevations  $(H_{\text{min}})$ , based on the same DEM. The *TRI* is therefore calculated as:

$$TRI = \sqrt{|H_{\text{max}}^2 - H_{\text{min}}^2|} \tag{2}$$

TRI = Terrain Ruggedness Index  $H_{max}$  = maximum elevations  $H_{min}$  = minimum elevations

For both methods, the continuous colour map was used in ArcGIS to represent the data visually, to avoid classification into artificially made categories. Prior to selection of an optimal size range ( $3 \text{ m} \times 3 \text{ m}$ ), we tried to produce different size ranges ( $1 \text{ m} \times 1 \text{ m}$ ,  $10 \text{ m} \times 10 \text{ m}$  and  $50 \text{ m} \times 50 \text{ m}$ ). The original size of  $1 \text{ m} \times 1 \text{ m}$ (high resolution) gave results which were too fragmented, and had excessive noise due to the network being too dense. In contrast, by choosing larger areas ( $10 \text{ m} \times 10 \text{ m}$  and  $50 \text{ m} \times 50 \text{ m}$ ) (lower resolution), the border was too blurred and the results again were not suitable for any morphometric analysis.

#### **Results and discussion**

According to the lithological diversity of the carbonate gravel (which belongs to the sedimentary body of the paleo-landslide of Sveta Magdalena) and of flysch base rocks (in the neighbourhood) the results of the slope variability and TRI were very useful. It turned out that the carbonate gravel of the sedimentary body has high slope variability and TRI values, which means that the sedimentary body of carbonate gravel exhibits a high degree of surface roughness. In contrast, the area made of flysch base rocks in the vicinity of the sedimentary bodies represents values of lower slope variability and TRI. Consequently, associate flysch represents an area with low surface roughness, that is, the surface in this part of the area is relatively smooth (Figure 3). Even from the shaded digital elevation model produced from

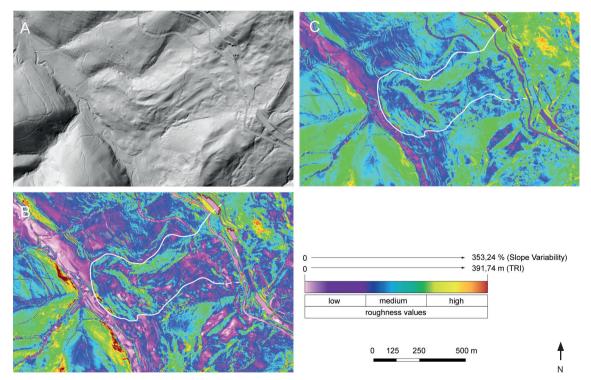
(1)

Lidar data (Figures 1 and 3A) it can be seen that the studied sedimentary body of the paleolandslide Sveta Magdalena and the immediate surroundings represent a range of evidently variable surface roughness.

#### Slope Variability

The results of the slope variability method are visible in Figure 3B, representing the difference between the highest and the lowest elevations. The casts of colours were divided roughly into three levels: low, medium and high variability of slopes. The casts of light to dark pink correspond to smooth surfaces (e.g., the Razdrto–Vipava motorway; upper (NE) part of the slopes), the cast of blue-green colour correspond to intermediate values (between smooth and rough surfaces), and the cast of yellowbrown colours correspond to areas with rough surfaces. Colour visualization was found to be useful to illustrate the areas with low and/or high slope variability.

The case of the paleo-landslide of Sveta Magdalena shows that the values of slope variability on the northern, western and southern parts of the sedimentary bodies are extremely high. High values of slope variability can be identified only in the lower part (fan-shaped part) of the sedimentary body at the toe of the landslide, while in the upper part, just below the carbonate Nanos massif, carbonate gravels in the form of scree deposit prevail, therefore a more accurate separation of this part of the sedimentary body is not possible. At the edge of the sedimentary body, blue-green colour casts prevail. At the toe of the landslide, yellow-brown colour casts that show medium to high surface roughness occur. These are in sharp contrast with the light-pink and pink colours which represent a smooth surface. Sharp transitions of slope variability on the outskirts of the sedimentary body can be attributed to steep margins and correspond to the lithological boundary, which represents the boundary of the Sveta Magdalena paleo-landslide. The sedimentary body composed of carbonate gravel mostly represents an area with medium to high surface roughness, while the flysch rocks mainly comprise smooth areas with very low slope variability (Figures 3B and C).

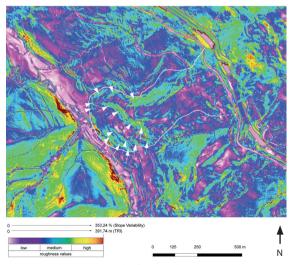


**Figure 3:** Two sample output for surface-roughness calculation with (A) digital elevation model of the Sveta Magdalena paleolandslide. (B) Slope Variability. (C) Terrain Ruggedness Index (TRI).

Higher slope variability at the edge, and in particular at the forefront, of sedimentary bodies can be explained by the phenomenon of inverse graded clasts and blocks in this part of the landslide. Typically, the debris flows of the larger particles move towards the edge of the flow and in a direction vertical to the surface current.<sup>[21, 22]</sup> This may result in inverse particle size classification and consequently, greater slope variability.

In the middle part of the fan-shaped sedimentary bodies on the surface of the individual bands, which are approximately parallel to the edge (toe) of the landslide, we recognized medium to high slope variability (arrows in Figure 4), which may indicate the individual reefs. Major<sup>[21]</sup> suggested, on the basis of experimental studies, that debris-flow deposition and deposits can be affected by degree of water saturation. As water saturation can affect debris-flow characteristics and deposition, clearly unsaturated flows exhibit steep margins and guite equant shapes with a width/length ratio greater than 0.5. Several arcuate ridges can appear on the surface due to successive waves of flow overriding and shoving the debris to the flume mouth.

Furthermore, it is obvious that in the fan-shaped sedimentary body two major areas emerged where the slope variability is extremely low (areas marked in Figure 5A). Areas with low slope variability are separated by an approximately

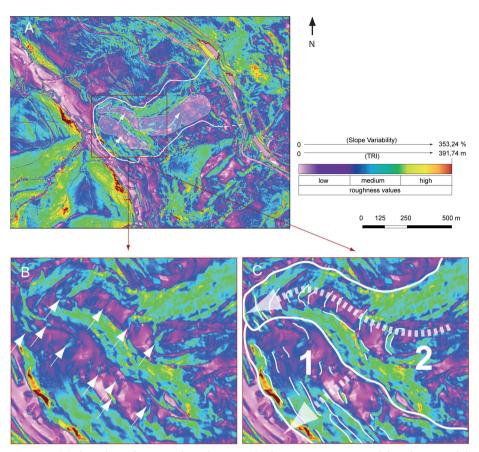


**Figure 4:** The arrows on the figure indicate individual separate bands with medium to high slope variability that are approximately parallel to the edge of the sedimentary body of the Sveta Magdalena paleo-landslide.

400 m long strip of high slope variability. These two areas, which represent a small surface roughness and are separated by areas of high surface roughness, could belong to two different sedimentary bodies. If these two areas are analysed in more detail, it can be seen that, even within areas with low roughness, smaller areas appear with a relatively high surface roughness (arrows in Figure 5B). This surface roughness could be attributed to the arcuate levees in smaller dimensions, which appear on the surface. From the forms of the arcuate levees with high slope variability, the direction of the sediment transport stream (Figure 5C) can be supposed. On the basis of the two different orientations of the arcuate levees on the surface of the sedimentary bodies (within an area with a relatively small variation in the inclination) we can conclude that the Sveta Magdalena paleolandslide, at least in the part of the fan-shaped sedimentary body, was most probably formed by two separate events, which were transported in the form of mass gravity flows<sup>[9, 10]</sup> down the slope in the Upper Vipava valley.

#### Terrain Ruggedness Index (TRI)

The results of the *TRI* are visible in Figure 3C. The cast of colours (similar to the slope variability) are divided roughly into three levels: low, medium and high TRIs. The cast of the light to dark pink colour corresponds to a smooth surface, blue-green colour tones correspond to intermediate values (between smooth and rough surfaces), and yellow-brown colour tones correspond to areas with a rough surface. Both methods give very similar maps (Figure 3), so the results for the TRI method can be similarly interpreted as the slope variability method. For this reason, only slope variability maps are shown in Figures 4 and 5. The latter method visually produces a larger range of data and is therefore preferred due to the more pronounced differences in relief roughness. One must, however, note that such a conclusion is based only on our study area and must be tested elsewhere. The results of both methods show that the surface roughness is the key variable in identifying the different forms of the surface and for the determination of the processes that have an effect on it.



**Figure 5:** (A) The indicated areas in the sedimentary body represent an area with low slope variability. (B) The arrows show individual and small separated areas with medium to high slope variability. (C) Approximate direction of transport of carbonate gravel, in the form of two debris flows (1: older and 2: younger) transported into the lower-lying parts of the Upper Vipava valley.

#### Conclusions

It has been shown that the surface roughness in this part of the Rebrnice area primarily depends on the properties of the material (lithology). The findings are similar to those of Grohmann et al. who, in addition to material properties, have attributed varying surface roughness to the current and past processes, and to the time elapsed since the formation of the surface.<sup>[23, 24]</sup> The high contrast between the high degree of surface roughness of the sedimentary body of the Sveta Magdalena paleo-landslide and the smoothness of the surface belonging to the area near the sedimentary body represent two different lithological units: the carbonate gravel, which belongs to the sedimentary body of the Sveta Magdalene paleo-landslide and flysch rocks, which constitute the bedrock and are today in the base and in the immediate vicinity of the paleo-landslide.

With the visual interpretation and the field validation, we achieved greater consistency in determining the shape of the relief structures and their metric properties. The properties of the surface of the sedimentary body of the Sveta Magdalena paleo- landslide in the Rebrnice area were identified by a visual interpretation of the digital elevation model and with calculated indicators of surface roughness. With both methods, slope variability and the TRI, we have recognized typical morphological characteristics expressed in the mass gravity flow. With visualization (GIS symbology), we could recognize lobate and fan-shaped bodies, increased surface roughness at the edges of the sedimentary bodies (steep margins), marginal levees and arcuate levees in the middle of the sedimentary body. In addition, we identified in part of the fan-shape body, in an area with relatively low slope variability and low TRI, two groups of curving ridges in different directions. All of these identified elements in the sedimentary body of the Sveta Magdalena paleo-landslide suggest that the sediments were transported in the form of unsaturated mass gravity flow (debris flow)<sup>[21]</sup> (probably in at least two separate events in time).

#### Acknowledgments

The authors would like to thank Geodetski inženiring Maribor, d. o. o.,<sup>[25]</sup> for allowing us to use the Lidar point cloud of the analysed area for non-commercial use. We also gratefully acknowledge The Ivan Rakovec Institute of Palaeontology, Scientific Research Centre SAZU, for their support and help.

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### Heavy metal geochemistry of stream sediments from parts of the Eastern Niger Delta Basin, South-Eastern Nigeria

Geokemija težkih kovin v rečnem mulju delov Vzhodne kadunje Nigrove delte v jugovzhodni Nigeriji

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#### Abstract

This study documents the baseline geochemistry of sediments in portions of the Eastern Niger Delta, focussing on the distribution of selected heavy metals (Pb, Zn, Cd, Cr, As and Ni). Concentration levels of metals were determined through Atomic Absorption Spectrophotometry (AAS) and results interpreted with statistical techniques. Interrelations between parameters reveal a control by three dominant geogenic factors. The study shows that heavy metal geochemistry of sediments is related to regional and local geology with minor imprints of anthropogenic activities.

**Key words:** trace metals, sediments, factor analysis, geogenic, Niger Delta, Nigeria.

#### Izvleček

Predmet študije je ugotovitev splošnih geokemičnih značilnosti rečnega mulja v nekaterih delih delte vzhodnega Nigra s poudarkom na porazdelitvi izbranih težkih kovin (Pb, Zn, Cd, Cr, As in Ni). Vsebino kovin so določili z atomsko absorpcijsko spektrofotometrijo (AAS) in rezultate interpretirali s statističnimi metodami. Medsebojne povezave med spremenljivkami nakazujejo odločilen vpliv treh prevladujočih geogenih dejavnikov. Rezultati raziskave dokazujejo, da je geokemija težkih kovin v rečnem mulju povezana z regionalno in lokalno geologijo ter z lokalnimi vplivi človekovih dejavnosti.

Ključne besede: sledne prvine, rečni mulj, faktorska analiza, geogeni vpliv, Nigrova delta, Nigerija

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#### Introduction

Concentration of heavy or trace metals within geo-environments have received tremendous attention in many research works world-wide;<sup>[1-5]</sup>. These have been predicated on a variety of interest including but not limited to the following; (a) their insidious contributions as related to public health concerns (b) their effects on biota growth and diversity and (c) academic interest in establishing baseline data and decipher the fate and dynamics of metal transport within geo-systems.

Chester et al.<sup>[6]</sup> conclude that coastal sediments are particularly important traps for trace elements.<sup>[1]</sup> observed that marine sediments in coastal regions near large industrial and urban areas contain heavy metals sometimes in amounts higher than their natural background. More than 90 % of transport of most heavy metals in river systems is shown to be as a solid phase in sediments<sup>[7]</sup>. Mobilization of these sediments into the major rivers and eventually into reservoirs may concentrate metals at nonbackground levels.

These metals exist as chemical species or fractions that exhibit different bioavailability and potential risk to human beings. These species and their character have been assessed successfully through statistical and geochemical modelling computer programs<sup>[8-11]</sup>.

This study attempts to document the heavy metal chemical trend and species availability of sediments in parts of the Eastern Niger Delta basin, south-eastern Nigeria. The natural geochemistries of sediments within the study area are believed to be controlled by geologic rock units through which rivers and streams within the catchment flow. High concentrations of heavy metals may be derived from mineral deposits, rock types with heavy metal concentrations above average crustal rocks as well as from weathering processes that may concentrate metals from rocks which do not contain large amounts of these metals. Contributions from anthropogenic activities may also be imprinted on the geochemical data of the sediments.

#### Description of study area

The area being described as the Eastern Niger Delta extends eastwards from the shoreline fringe of Ikot Abasi area to the coastal plain physiographic provinces of Calabar area of south-eastern Nigeria. The basin is about 100 km long and about 25 km wide, delimited by longitudes 7° 30'-8° 15' E and latitudes 4° 30'-4° 40' N (Figure 1). The basin spans the shore lines of Akwa- Ibom and Cross River States of south-eastern Nigeria.

Geologically the study area is composed of Tertiary and Quaternary sediments referred to as Coastal Plain Sands of the broader Niger Delta basin<sup>[12]</sup>. This formation consists of alternating sequence of gravel, sand, silt, clay and alluvium. Sediment fill within the basin are sourced from three major geologic units on the hinterlands. These units include;

- The Precambrian Oban Massif Complex made up of migmatites-gneisses, granites, schists, para-schists, pegmatites and a host of other ultra-mafic rock suites,<sup>[13]</sup>
- Cretaceous sedimentary fill known as the Calabar Flank, composed of limestones, sandstones, shales and marls<sup>[14]</sup> and
- The lower Benue Trough (Anambra Basin) of post-Cretaceous sediments with lodes of sulphide deposits.<sup>[15]</sup>

Another source of sediments into the study area is the Cross River Delta (Rio Del Rey Basin).<sup>[14, 15]</sup>

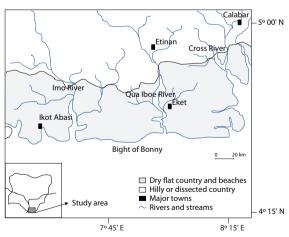


Figure 1: Map of Niger Delta Basin showing sampled rivers; inset index map of Nigeria (modified from Elueze et al., 2009).

w(Zn)/

Heavy metal geochemistry of stream sediments from parts of the Eastern Niger Delta Basin, South-Eastern Nigeria

Three major rivers (Imo, Qua-Iboe and Cross River) drain the geologic units of the hinterlands alongside their lesser tributaries of other rivers and streams.

Morphologically the area lies within the flat and low-lying terrain of the coastal lowland/ Niger Delta region of southern Nigeria with elevations ranging from less than 10 m at the coastal fringe to about 80 m northwards.

The study area receives an average rainfall of about 254 mm annually within two distinct seasons; dry and wet seasons. Mean annual air temperature and relative humidity are 26.8 °C and 84.6 % respectively.<sup>[16]</sup>

### Methodology

**River**/

A total of 52 stream sediment samples of about 1.0 kg were collected from the three major rivers and their tributaries within the study area. The samples were collected by hand held au-

w(Pb)/

ger and hand scooping at shallow depths of
0-30 cm, then stored in clean labelled poly-
thene bags prior to treatment and analysis.

The sediments were air dried and disaggregated in a porcelain mortar using a rubberend pestle. A nylon sieve was used to obtain the < 63  $\mu$ m fraction of the samples for further chemical analysis. Element content analyses of replicate samples were carried out by Atomic Absorption Spectrophotometry (AAS) at the Activation Laboratories Ltd., Canada and locally in the quality control laboratory of the Aluminium Smelter Company, Nigeria. Results indicated good coincidence of data and reliability of analyses.

#### **Results and discussion**

w(Cr)/

Table 1 shows a statistical summary of the trace element concentrations of stream sediments sampled from the study area. The table

w(As)/

w(Ni)/

	"(= ~)/	·· ()/	"("")	"(")	"("")/	<i>"</i> ()/
Element	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
<b>CRS</b> ( <i>n</i> = 20)						
Range	27.40-86.40	88.30-117.85	1.20 - 2.60	0.10 - 1.70	1.60 - 2.20	1.20-14.60
Mean	56.51	108.77	1.85	0.97	2.05	7.03
S. D.	22.81	12.90	0.44	0.47	0.93	4.50
<b>QIR</b> ( <i>n</i> = 16)						
Range	38.20-68.35	74.20-108.30	1.00-3.20	0.50-1.20	1.30-3.80	1.20-8.40
Mean	53.12	90.55	1.78	0.82	2.56	5.44
S. D.	10.11	11.87	0.72	0.22	0.85	2.15
<b>IMR</b> ( <i>n</i> = 16)						
Range	9.00-29.00	31.00-306.00	1.00 - 1.80	10.00-67.00	30.02-22.00	6.00-28.00
Mean	18.81	65.90	1.40	40.06	6.88	15.73
S. D.	14.14	194.45	0.57	40.31	5.66	15.56
Bight of	16.41	647.6	0.71	16.71		
Bonny	(7.20-45.5)	(180 - 1410)	(0.30 - 1.60)	(6.40 - 46.10)	-	-
Calabar River	(0.60-30.0)	(0.80-27.0)	-	(0.60-3.30)	_	(1.20-22.50)
Gulf of California	17	88	3	44	7	38
Average shale	20	95	1	90	10	68

w(Cd)/

Table 1: Statistical summary of trace metal concentrations in sediments

CRS – Cross River sediments QIR – Qua Iboe River sediments

S. D. – Standard deviation

Calabar River<sup>[4]</sup> Gulf of California<sup>[18]</sup> IMR – Imo River sediments

Average shale<sup>[19]</sup> \* values in parentheses indicate ranges

also shows global averages as well as data of studied sediments of similar provenance as a means of comparison.

The trend of dominance of the trace elements for the Cross River and Qua Iboe River sediments was Zn > Pb > Ni > As > Cd > Cr while for Imo River the trend was Zn > Cr > Pb > Ni> As > Cd.

Lead (Pb), zinc (Zn) and cadmium (Cd) showed highest mean values from the Cross River, followed by Qua-Iboe and least values in Imo River sediments. On the other hand chromium (Cr), arsenic (As) and nickel (Ni) had highest mean values on the Imo River sediments and lesser in the other two rivers. Mean variances of data from the three rivers were assessed using t-test. At 95 % confidence level values are: CRS/QIR (0.012), CRS/IMR (0.036) and QIR/ IMR (0.047). These indicate no significance of variance of data between the rivers. Higher values of Pb. Zn and Cd in the Cross River can be attributed to the catchment of the river. This is mainly the Precambrian basement dominated by ultra-mafic and mafic rock suites that are hosts to most trace and rare metals species as revealed from hydrogeochemical modelling<sup>[10]</sup>. Economic activities of mining and quarrying within the basement complex are also responsible for disaggregation and mobilization of sediments with species of these metals. Cross River and Qua Iboe rivers drain most of the urbanized and industrial settlements within the study area. These areas have the presence of large and small scale processing and production outfits, automobile repair and service shops and wide variety of artisanal industries, which may serve as sources of trace metal release from their effluents into the environment. Agriculture is the main occupation of the inhabitants of the study area and possibly past practice resulting in erosion and deposition of sediments within stream channels have affected and may continue to influence stream sediment geochemistry.

Higher Cr, As and Ni concentrations in the Imo River are attributable to the shale dominated sedimentary geology of the lower Benue trough (Anambra basin).

Mean values of heavy metals for sediments from the entire study area were; Pb (34.30 mg/kg), Zn

(80.00 mg/kg), Cd (1.55 mg/kg), Cr (23.10 mg/kg), As (3.90 mg/kg) and Ni (11.60 mg/kg).

Observation reveals the heavy metals to be within ranges and in proximity to levels of sediments of similar provenance and global averages. The values are close to background and below levels that can affect the quality of life of marine biota.<sup>[3]</sup>

#### Factor analysis

Correlation and factor analyses were executed on the heavy metal concentration of sediments in order to identify important and significant interrelations. The extraction method for factor analysis was based on the maximum likelihood factors. Correlation matrix (Table 2) shows that there exists a strong positive correlation between Cr and Ni (0.86). A moderate negative correlation is seen between Cr and Pb (-0.66). Correlations among all other elements are generally weak (<  $\pm 0.50$ ). Variations in geochemical mobilities of the metals result in a non-uniform distribution pattern with a cumulative distribution function of 0.006.

The raw and unrotated data was subjected to factor analysis (Table 3) and this extracted three factors accounting for over 82 % of total data variance. Cluster of loadings shows that Pb (0.59), Cr (0.93) and Ni (0.93) for factor 1 and Zn (0.67), Cd (0.67) and As (0.72) for factor 2 determines the oblique factors for hierarchical analysis. Only factor loadings with scores greater than  $\pm 0.60$  were considered significant and are marked in the factor table.

Factor 1 (Pb, Zn and Cd) extracted about 49 % of total data variance. The loadings reflect signatures of sediments derived from the weathering of sulphide lode deposits found within the lower Benue trough. There is a reported occurrence of galena (PbS) and sphalerite (ZnS) in the Abaka-liki area of the lower Benue trough and this area forms part of the drainage basin of the eastern Niger Delta.<sup>[20, 5]</sup>

Factor 2 (Cr and Ni) accounts for about 20 % of total data variance and represents inputs of sediments of ultra-mafic origin. This loading can be associated with lineament zones (faults and joints) which may contain sheared ultra-mafic rocks. Cr and Ni coincidence suggest association with meta-basaltic rocks (amphibolites),<sup>[21]</sup> and these are common within the adjoining base-

ment complex. Effects of acidic rain water leaching altered rocks of the basement complex and Cretaceous rocks of the Calabar Flank can also be adjudged.<sup>[22]</sup> Sandstone has been shown to be a major source of trace elements<sup>[3]</sup>, thus supporting the role of the Cretaceous Awi sandstones of the Calabar Flank.<sup>[5]</sup>

Factor 3 (As), accounts for about 12.5 % of total data variance. This factor suggests litho-geochemical input representative of weathering and mobility of sediments from arsenic or related mineralisation. Cretaceous sediments as well as the poly-metallic sulphide lodes of the lower Benue trough have been reported of Mo-As associations.<sup>[23]</sup>

Table 2: Correlation matrix for heavy metals

	Pb	Zn	Cd	Cr	As	Ni
Pb	1					
Zn	0.29	1				
Cd	0.38	0.37	1			
Cr	-0.66	-0.45	-0.38	1		
As	-0.24	-0.23	-0.25	0.22	1	
Ni	-0.33	-0.14	-0.20	0.86	0.03	1

Parameter	Factor 1	Factor 2	Factor 3
Pb	0.708	-0.216	0.034
Zn	0.736	0.285	0.510
Cd	0.688	0.540	0.203
Cr	0.563	0.649	0.421
As	0.535	0.533	0.606
Ni	0.411	0.837	0.116
Eigen value	2.959	1.213	0.753
Total variance (%)	49.311	20.226	12.543
Eigen value cumul.	2.959	4.172	4.925
Cumul. variance (%)	49.311	69.537	82.081

#### Conclusion

This study has provided an important baseline geochemical data for the eastern sector of the Niger Delta basin. The heavy metals are generally at levels within or in proximity to global background levels and sediments of similar provenance. The data indicates that regional and local geology are the most important factors controlling heavy metal geochemistry. Possibly localized anthropogenic activities may affect stream sediment geochemistry. Statistical analysis reveals interrelations between metals as indicated by factor loadings to be controlled by three dominant factors of geogenic origin.

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## Profiling and analysis of the overconsolidation ratio and strength parameters in hungarian soils of the metro 4 stations in Budapest, Hungary

Profiliranje in analiza prekonsolidacijskega količnika in trdnostnih parametrov v kiscellijski glini za postaje proge 4 metroja v Budimpešti na Madžarskem

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#### Abstract

The study is about the determination of the overconsolidation ratio and the strength parameter analysis related to Metro 4 stations in Budapest. The overconsolidation ratio was determined from oedometer tests using the Casagrande method, and from cone resistance of Cone Penetration Tests (CPTu) in the upper soil strata. For the metro stations a great number of boreholes were made with core samples carried out from depths of 10 m to 40 m. Unloading-reloading modulus ( $E_{ur}$ ) and secant modulus at 50 % strength ( $E_{50}$ ) were derived from triaxial tests carried out with the mentioned layer called Kiscell clay in the Buda side. In this clay, the strength and consolidation parameters dominantly depend on depth. In this paper the correlation between the different parameters were determined.

**Key words:** overconsolidation ratio, CPTu, oedometer test, triaxial test, Kiscell clay

#### Izvleček

Predmet študije sta bila določanje prekonsolidacijskega količnika in analiza trdnostnih parametrov pri gradnji postajališč proge 4 podzemne železnice v Budimpešti. Prekonsolidacijski količnik so določali z edometrskimi preizkusi po Casagrandejevi metodi in odpornostjo stožcev pri preizkusu s statičnim konusnim penetrometrom (CPTu) v zgornjih plasteh zemljine. Na lokacijah postaj metroja so zvrtali večje število vrtin, ki so jih jedrovali v globinah med 10 m in 40 m. Modul razbremenitve-ponovne obremenitve  $(E_{m})$  in sekantni modul pri 50-odstotni trdnosti  $(E_{50})$  so izpeljali iz rezultatov triosnih preizkusov, ki so jih izvedli v hribini s kiscellijsko glino na območju Bude. V preiskovani glini so trdnost in konsolidacijski parametri izrazito odvisni od globine. V članku je opisano ugotavljanje povezave med različnimi parametri.

Ključne besede: prekonsolidacijski količnik, CPTu, edometrski preizkus, triosni preizkus, kiscellijska glina

#### Introduction

For the construction of Metro Line 4 in Budapest, a large number of soil explorations in high quality was required, and through the processing of these samples, further information have been gathered about the clay soils situated in great depths, and on the changes of the relevant soil-physical parameters in the function of depth. Triaxial and oedometer tests were carried out by using continuous core output samples.<sup>[1]</sup> For the soil models of advanced computer softwares using finite-element models (e.g. the hardening soil model), a knowledge of the soil parameters is rather important in order to ensure that the modeling should approximate reality as much as possible. In this paper the analysis and profiling of the strength and overconsolidation parameters of soil strata, and the relationship among these parameters are presented.

# Stress history and overconsolidation ratio

#### **Oedometer test**

One-dimensional oedometer tests are suitable for determining the oversonsolidation ratio (*OCR*) and the consolidation parameters. The yield point denotes the preconsolidation stress ( $\sigma_p$ '). Determination of  $\sigma_p$ ' from void ratio  $e - \lg \sigma$  relationship is presented in Figure 1, where  $\sigma$  is the loading stress in kPa.<sup>[2]</sup>

In normalized form, the degree of preconsolidation is called the overconsolidation ratio (Equation 1).

$$OCR = \frac{\sigma'_p}{\sigma'_{y0}} \tag{1}$$

where  $\sigma_{v0}$ ' is the effective vertical geostatic stress. If *OCR* is less than 1, under-consolidated, if equal to 1, normally consolidated, and if greater than 1, then we are talking about over-consolidated soil. *OCR* is defined as the highest stress experienced divided by the current stress.

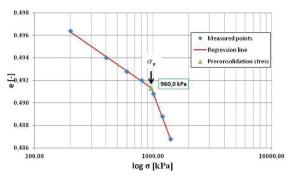


Figure 1: Determination of preconsolidation stress.

#### Cone Penetration Test (CPTu)

CPTu test results were compared with the results obtained from further in-situ Self boring pressuremeter (SBP) tests analysed by E. Kalman (2012) and laboratory tests. CPTu is one of the most frequently used in-situ tests in Hungary. These test methods are suitable for soil classification and derive soil-mechanical properties. The test method can be used easily and efficiently.

#### Triaxial tests

Reloading modulus ( $E_{\rm ur}$ ) and secant modulus at 50 % strength ( $E_{\rm 50}$ ) were determined from triaxial tests using large diameter (10 cm) specimens from depths of 10 m to 40 m.<sup>[1]</sup> The correlation between these parameters and depth was analysed.

#### **Measurement results**

On the basis of the tests, a good approximation can be achieved in determining the *OCR* value (that changes in the function of depth) by using power functions. The oedometer tests were carried out by using samples taken in depths between 5 m and 45 m, and then the determined *OCR* values were depicted in the function of depth values (see Figure 2). The results of 142 samples from 11 sites were used for the function.

The extent of approximation shows that it is not possible to handle all the stations together, however it reflects the *OCR* values properly: while depth grows, the *OCR* values get lower and move within a given range. This justifies the necessity of explorations to be taken, as well as analyses of the concomitant circumstances. For

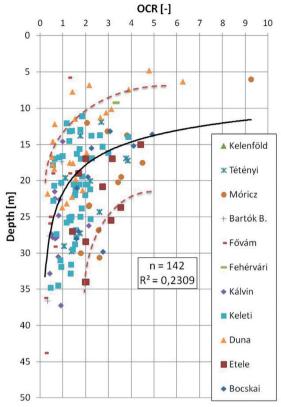


Figure 2: OCR value versus depth value.

the values belonging to some of the stations, a matching of functions took place, which is summarized in Table 4. The broken lines mark interpretation domains (see Figure 2). The Clay is from normal to heavily overconsolidated, its overconsolidation ratio varies between 0.2 and 9.3 depending on depth. Where the OCR value is less than 1, the soil is underconsolidated. In this case it could be caused by the fact that the given level was measured at loading stress in the test, and the effective stress was higher, therefore the yield point showed a local point and not the global location in the  $e - \lg s$  curve. Lower value of loading stress of oedometer test than it is necessary (for example only 800 kPa) can be resulted lower preconsolidation stress.<sup>[3]</sup> A lower than 1 value is not likely, so it can be established that laboratory tests are sensitive to the preparation of samples, the time lapsing between the test and the sampling event, as well as the level of the loading stress during the test. Higher loading stress could be resulted more accurate yield point to define preconsolidation stress. For increasing the accuracy of the approximative functions, several in-situ and

laboratory measurements are needed, in addition to the fact that the inhomogenity of soils is pointed out by the standard deviation of the values.

Harder soil strata cannot be penetrated for using a CPTu test, therefore this method is sufficient only to reach the upper, weathered layers of the Clay. In the event of Fővám tér, Bocskai út and Kelenföld stations, CPTu test results, while in the event of Fővám tér and Bocskai út stations, oedometer test results have been compared with the results of SBP tests that had been performed earlier in this soil layer.<sup>[4]</sup>

*OCR* can be expressed by the Equation 2 using CPTu Test, Powell et al. (1988):

$$OCR = k \cdot \left(\frac{q_t - \sigma_{v_0}}{\sigma'_{v_0}}\right) \tag{2}$$

where k is the overconsolidation factor and  $q_t$  is the corrected cone resistance.<sup>[5]</sup> For the upper soil strata k = 0.2 was used.

The *OCR* values based on CPTu, laboratory and SBP tests are presented in Figure 3.

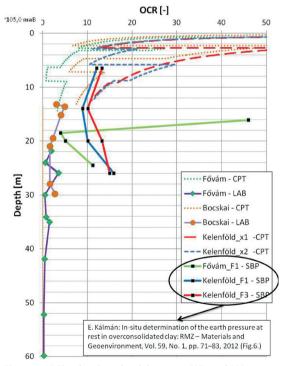
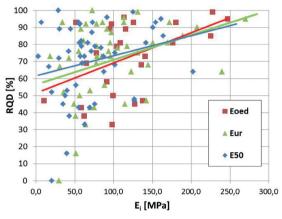


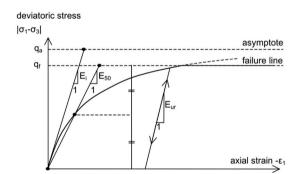
Figure 3: OCR values based on laboratory, CPTu and SBP tests.

Further on, a statistical analysis of the stiffness parameters comes. The available Rock Quality Designation index (RQD) values determined on the basis of the borings belonging to Kálvin tér, Móricz and Etele stations have been compared with the  $E_{\rm ur}$  and  $E_{\rm 50}$  values of the oedometer modulus ( $E_{\rm oed}$ ). There is a high standard deviation between the values, but it can be seen that the modulus values grow parallel with the improvement of the *RQD* value, as shown by Figure 4.



**Figure 4:** RQD vs.  $E_{oed}$ ,  $E_{ur}$  and  $E_{50}$ .

In the Hardening Soil model (Plaxis) the relationship is hyperbolic between the vertical strain,  $\varepsilon_1$ , and the deviatoric stress, q, in the primary triaxial loading (see Figure 5).

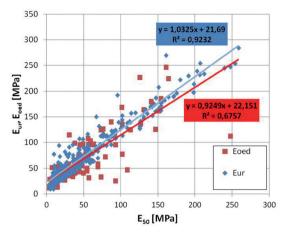


**Figure 5:** Hyperbolic stress-strain relation in primary loading for a standard drained triaxial test [Plaxis Manual].

As the Plaxis software recommends,  $E_{ur}$  should take the value of  $3 \times E_{50}$ , but the measurements show that they rather approximate the value 1. The relationships among the three moduli are shown by Figure 6.

Figures 7, 8 and 9 depict the changes of the  $E_{oed}$ ,  $E_{ur}$  and  $E_{50}$  values in the function of depth, and their approximation to the straight line, where

the results of the stations of Buda side involved in the research are shown.



**Figure 6:** Correlation between  $E_{50}$  and  $E_{ord}$ ,  $E_{ur}$ .

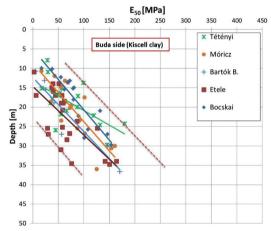


Figure 7: E<sub>50</sub> value versus depth.

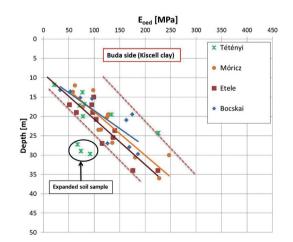


Figure 8: E<sub>ord</sub> value versus depth.

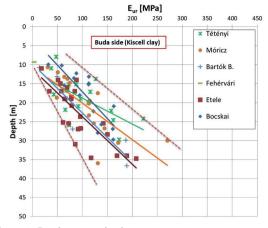


Figure 9: E<sub>m</sub>value versus depth.

By handling the various stations separately, the moduli and the changes of *OCR* versus depth have been determined by using the following correlations (Equation 3, 4);<sup>[6]</sup> then Tables 1–4 give a summary of the values of the variable factors (a, b, A,  $\alpha$ ), the approximation ratio of the approximative function ( $R^2$ ) and the number of data points (n).

$$E_i = a \cdot (z) + b \tag{3}$$

$$OCR = A \cdot (z)^{\alpha} \tag{4}$$

where *z* is the depth and it should be interpreted by using the meter dimension.

Table 1:  $E_{so} - z$ 

Location	а	b	$R^2$	n
Tétényi	13.28	-177.36	0.40	18
Móricz	6.68	-58.50	0.66	16
Bartók B.	7.18	-88.74	0.88	7
Etele	7.96	-116.55	0.51	24
Bocskai	8.63	-44.04	0.78	15

Table 2:  $E_{ur} - Z$ 

Location	а	b	$R^2$	n
Tétényi	14.60	-174.34	0.40	18
Móricz	10.37	-108.66	0.57	16
Bartók B.	7.72	-75.23	0.86	7
Etele	7.97	-88.86	0.65	24
Bocskai	7.10	-21.84	0.69	15

**Table 3:**  $E_{oed} - Z$ 

Location	а	b	$R^2$	n
Móricz	8.87	-66.72	0.80	14
Etele	8.49	-75.81	0.78	11
Bocskai	10.78	-99.45	0.67	9

**Table 4:** *OCR* – *z* 

Location	A	а	$R^2$	n
Tétényi	3683.33	-2.39	0.40	3
Fővám	31.89	-1.33	0.65	8
Duna	773.95	-2.42	0.44	22
Etele	550.75	-1.68	0.58	7
Bocskai	389.69	-1.74	0.70	7

The negative values of parameter *b* indicate that there was no measurement in the upper 10 m zone, as it belongs to a different soil type, therefore the approximative straight line determined by the statistical analysis is valid from cca. the 10 m depth on, and this is true also for the *OCR*-depth correlation and approximative function.

## 4) **Conclusions**

Taking into consideration first the unloading then the reloading of soils in the event of deep excavations, a good approximation can be achieved for such behaviours of soils by using the Hardening Soil Model offered by the finiteelement softwares widely spread in practice. For the description of the behaviour of the Kiscell Clay occurring frequently in Buda side and of Tardi Clay typical of Pest side, the solution would be the construction of a new soil model. but the designers of the Metro construction project had decided in favour of the Hardening Soil Model mentioned above. For this application, "special" input soil parameters were needed such as  $E_{50}$ ,  $E_{ur}$ ,  $E_{oed}$ , and OCR. These parameters have been depicted in the function of the changes of depth so that a picture should be drawn up on the nature of changes. Occasionally, the high standard deviation and low correlation values of the approximative functions were determined by the inhomogenity of soils or (just in the course of testing) by the measureability range of deformations, or by the condition and preparation (and preparation-ability) of soil samples. In specifying the preconsolidation stress values of soils, the preparation and carving of samples, the maximum loading stress selected, and waiting out while consolidation takes place have especially high effects, as well as the time lapsing between the taking and the testing of samples, and further important but not vet examined or less examined factors, such as temperature, are also conceivable. In filtering the data, the taking into consideration of the categories of RQD did not yield better approximative functions, but as regards layers, efforts have been taken for testing soils of the same classes.

In general, the calculated Overconsolidation ratio decreased due to depth, depended on the determination method. The determination of *OCR* can be based on laboratory, CPTu or SBP tests, as well. The upper 3–5 m thick layer can result in high values of *OCR* by using the CPTu method, because the effective vertical stress is low in the upper soil strata and the cone tip resistance can be high.<sup>[3, 6]</sup> Therefore, these high values in the upper layers should not be used in soil models of finite element modeling methods.

Lower value of loading stress of oedometer test can be resulted lower preconsolidaton stress than the derived values from in-situ test. Therefore, higher loading stress (1 400–2 000 kPa) could be used to more accurate preconsolidation stress.<sup>[3]</sup> It is recommended that the loading stress should be used up to a  $2-3 \times$  value of the total stress in order to ensure a clear outlining of the yield point by using the Casagrande method. Where the purpose is to determine the overconsolidation ratio of soils by using oedometer tests, more accurate values can be received by using loading stresses higher than the stress level belonging to them, beside the assumed overconsolidation ratio.

*OCR* values based on oedometer, CPTu and SBP test results were compared using previous research results.<sup>[4]</sup> It is recommended to perform the in-situ tests parallel with the laboratory tests, because a more accurate approximation can be given on the stiffness and pre-loading

behaviour of soils by using higher amounts of data.

The determined strength parameters, as well as their correlation with depth and with one another open the way for the future for performing back-analyses, and permit the design and dimensioning of construction projects in large depths, similar to the Metro 4 project in Budapest to be carried out in an economic manner.

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Problematika ugotavljanja sedanjih obremenitev okolja v Nigeriji

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#### Abstract

For development to be sustainable it should meet the emerging needs of the present and succeeding generations. It must focus on growth without compromising the needs of the future. Environmental engineering requires that the impact and interaction of common engineered structures such as dams, highways, deep foundation buildings, among others are carried out with minimal environmental deterioration. Agricultural practices, oil exploitation activities, mining operations and industrial waste disposal which affect our environment tremendously, should be subjected to comprehensive and adequate assessment of the impacts they will have on our environment before the implementation of such activities. This will lead to adequate preparation for effective mitigation of future environmental hazards that might accompany these activities.

**Key words:** sustainable development, environment, projects, future, hazard mitigation

#### Izvleček

Če naj bo razvoj trajnosten, moramo upoštevati naraščajoče potrebe sedanje in prihodnjih generacij. Usmerjen mora biti v rast, ki ne bo ogrožala potreb prihodnosti. Okoljsko inženirstvo mora skrbeti za to, da sta obremenitev in vpliv gradbenih objektov, kot so pregrade, ceste, globoko temeljene zgradbe, čim manj kvarna za okolje. Postopke v poljedelstvu, pridobivanje nafte, rudarjenje in odlaganje industrijskih odpadkov, kar drastično obremenjuje naše okolje, je treba ustrezno in izčrpno raziskati z vidika vplivov na okolje že pred začetkom razvoja teh dejavnosti. S takim ravnanjem se bo mogoče ustrezno pripraviti na učinkovito spopadanje s prihodnjimi okoljskimi tveganji, ki utegnejo spremljati te dejavnosti.

**Ključne besede:** trajnostni razvoj, okolje, projekti, prihodnost, grožnje okolju

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## Introduction

The consideration of the impact of engineered structures and developmental processes on the natural environment is important in any project. To achieve sustainable development in any activity in Nigeria, EIA is key tool for mainstreaming environmental concerns into development process. Nigeria is one of the few developing countries that have specific relevant legislation as enacted in the 1992 EIA act. This paper reviews some research works on the effects of some of the aforementioned activities on our environment and makes recommendations. There are points of differences between Nigerian systems and the more conventional EIA systems. Federal Environmental Protection Agency (FEPA) was also established in 1998 to control the Nigerian Environment, its resources exploitation and management. But field observation revealed that environmental degradation is growing at a rate worse than the pre FEPA period.<sup>[1]</sup> FEPA and other relevant Departments in other Ministries were merged to form the Federal Ministry of Environment in 1999. The National Environmental Standards and Regulations Enforcement Agency (NES-REA) was also established as a parastatal of the Federal Ministry of Environment, Housing and Urban Development in 2007. This paper examines the shortcomings in the Nigerian system and also discusses the present effects of these inadequacies.

#### The scenario

As the Nigerian population continues to increase, more people live in cities and towns. Agricultural land located near cities is being converted to suburban housing. As people populate areas that were once agricultural or rural, stores and industry follow. The development of urban areas has many environmental impacts. When towns and cities expand into rural areas, natural habitats are lost to roads, houses, and other buildings. Development leaves less land for agricultural use, which puts pressure on the remaining farmland for increased production. Other problems are created when concrete and asphalt cover large areas. Because there are fewer opportunities for rainwater to soak into the ground, groundwater supplies are not

recharged and flooding increases during heavy rains. This is evident in the increase in flood incidents in the Nigerian urban areas. Additional contamination occurs as a result of industrial processes. Heavy metals, such as lead and mercury, and poisonous chemicals, such as arsenic, are by-products of many industrial processes and can pollute the soil and groundwater. Some of this type of contamination has been caused by industries that operated without putting into consideration the possible environmental impacts potential dangers of improper waste disposal. In the United States, wetlands are now recognized as valuable ecosystems and are protected from development but in most of the Nigerian wetland resource especially in heavily populated urban areas are disappearing daily due to residential extensions and other construction operations.<sup>[2]</sup> Modern landfills are carefully designed to minimize leakage of toxic liquids. Impermeable clay or plastic layers are placed beneath a landfill, but most of the Nigerian waste disposal sites including government established landfill sites are currently posing health treats to their immediate environments.

#### Identified shotcomings

- There is delay in the execution of EIA in Nigeria due to the inadequacies and misinterpretations of various regulatory statutes.
- There is duplication of functions and overlapping responsibilities in processes and procedures guiding the execution of the various impact assessment tasks due to too many regulatory bodies (FME NESREA, NOS-DRA, DPR, states EBP).
- Absence of effective Sanctions.
- A large percentage of Nigerian populace is unaware of EIA provisions and their rights of objection to environmentally unfriendly prospective projects during the 21 days public display of EIA drafts.
- Abuse of the exclusion clause in the EIA act.
- Lack of credibility and transparency.

EIA in Developed Countries	EIA in Developing Countries	EIA in Nigeria
Well-framed EIA legislation in place.	Lack of formal EIA legislation in many Developing countries.	For legislation for EIA through the enactment of the EIA Act No. 86 (1992). Nigeria is the first African country to establish a national institutional mechanism for environmental protection.
In developed countries, active involvement of all participants including competent authority, government agencies and affected people at early Stages of the EIA. This makes the process more robust and gives a fair idea of issues, which need to be addressed in the initial Phase of EIA.	Limited involvement of public and government Agencies in the initial phases. This often results in poor representation of the issues and impacts in the report, adversely affecting the quality of the report.	Limited involvement of public and government agencies in the initial Phases.
The process of screening is well defined. For instance, in EU countries competent authorities decide whether EIA is required after seeking advice from developer, NGO and statutory consultees. In Japan, screening decision is made by the authorizing agency with respect to certain criteria.	In developing countries, screening practice in EIA is weak. In most cases, there is a list of activities that require EIA but without any threshold values.	Federal Ministry of environment does internal screening to determine the projects category under mandatory study activity list.
Scoping process is comprehensive and involves consultation with all the stakeholders. In many countries like US, Netherlands, Canada and Europe, the involvement of the public and their concern are addressed in the scoping exercise. Besides this, funding organizations such as World Bank, ADB and ERDB have provision for consultation with the affected people and NGOs during identification of issues in scoping exercise.	Scoping process in most developing countries is very poorly defined. In many countries including China, Pakistan, etc. there is no provision for scoping. In countries where it is undertaken, there is no public consultation during scoping. Moreover, in most developing countries, scoping is often directed towards meeting pollution control requirements, rather than addressing the full range of potential environmental impacts from a proposed development.	In Nigeria, a term of reference is followed for scoping. Proponent carry out EIA generally using consultants and drafts EIA copies is submitted to the responsible officer with an annex record of public form.
Most reports in local language.	Most reports in English and not in the local language.	All repots are in English.
Proper consideration of alternatives in EIA.	The consideration of alternatives in developing countries is more or less absent.	There consideration of alternatives is more or less absent.

A multi – disciplinary approach. Involvement of expertise in different areas.	Lack of trained EIA professionals often lead to the preparation of inadequate and irrelevant EIA reports.	EIA is carried out by consultants. Some have multidisciplinary teams while some do not. The level of expertise also vary widely within the system.
Two tier of EIA review. One conducted after the completion of EIA to check the effectiveness of EIA and the second done before decision making.	Poor review and monitoring.	There are three methods of review and public participation is not compulsory in all cases. Poor implementation and monitoring of EIA recommendations.

Modified after Govind<sup>[3]</sup>

## The effects of these inadequacies

## **Recurrent Failure of Earth Structures**

**Table 2:** Statistics of some collapsed buildings

Source	Date of occurrence	Place of occurrence	Number of casualties
Nigerian Tribune	June 16, 1990	Private Secondary, School, Saque Comprehensive College Diobu Area.	28
Daily Trust	June16, 1990	PortHarcourt, An extension of Boungalow 36, Obasiolu Street Mile 3, Diobu	54
Daily Tribune	July 6, 1990	Port Harcourt School Building, Diobu	100 students
National Concord	August 2, 1991	Lagos - Ipaja Ikotun - Isheri uncompleted building	10
Nigerian Tribune	Jan 5,1995	Lagos - Maryland	1
Daily Trust	July 20, 2006	Minacity, Lagos	25 dead, 50 survivors
Daily Trust	July 21, 2006	Alagbado, Lagos	23 dead, 52 survivors
Daily Trust	July 21, 2006	Mushin Olosha, Lagos	36 dead, 1 rescued
The Daily Punch	March 27, 2007	Lagos three storey building conctruction	11
Daily Ttrust	April 17, 2009	Abuilding under conctruction at Auchi	6
The Gaudian	August 11, 2010	3 - Storey building Okoli Street Area 11, Garki Abuja	17

Adapted from The Crust<sup>[4]</sup>

#### Incessant flooding

In Nigeria, flood affects and displaces more people than any other disaster; it also causes more damage to properties. At least 20 per cent of the population is at risk from one form of flooding or another.<sup>[5]</sup> Series of photos released by the National Emergency Management Agency (NEMA) shows the seriousness of flooding that devastated various parts of Nigeria during this raining season. Some 2 million Nigerians have been displaced from their homes while some have died or gone missing as a result of the ravaging flood.

The most striking aspect of the flooding happened on the ever-busy Abuja-Lokoja highway where the key highway linking northern Nigeria to the rest of the south, generally the Southwest was submerged under water for weeks, causing traffic delays that lasted days. Several residential buildings, schools, churches and hotels were submerged by the flood. Oye Ibidapo-Obe in a press briefing said "If a proper risk assessment was done the Lokoja flood disaster would have been mitigated even before the road from Lokoja to Abuja that was cut off was built". Lack of adequate Environmental impact assessment makes us unprepared for mitigation of natural disasters such as this.

## **Environmental Degradation**

Despite the legal backing and funding, which established institutions in charge of the environment enjoys from the federal government, their level of success in environmental protection and hazard mitigation is low. Omofonmwan and Osa-Edoh <sup>[1]</sup> attributed the situation to the fact that concepts and ideas this institutions operate with did not evolve from the people's tradition or way of life. They also predicted that if appropriate techniques and technology of environmental protection and management are not put in place, Nigeria may become a difficult country to live in the next 15 years.

In 1999, the United Nations declared the delta the most threatened in the world due to degradation from crude oil pollution.<sup>[6]</sup> Oil spillages have also caused depletion of the vegetation cover and the mangrove Ecosystem in this re-

S/No	Location	Associated hazard	Affected number of people	Date
1.	Adamawa State	Flood Houses & Farmlands destroyed	500	April 2001
2.	Akwa - Ibom State	367 houses washed away	4 000	March 2001
3.	Kano State	434 houses destroyed, one dead and 20 corpses unearthed	Not available	August 3013
4.	Lagos State	Buildings collapsed, markets submerged, propertries destroyed.	Over 300 000 affected	Early 1970's Till Date
5.	Taraba State	80 Houses totally swept off. 410 Houses extensively destroyed	More than 50 000 displaced	August 2005
6.	Sokoto State	Houses, food stores and farm lands were destroyed	49 dead, 130 000 displaced	September 2010
7.	Zamfara State	Building submerged, Farmlands destroyed, Properties damaged	12 398 affected	July 2001

Table 3: Some Episodes of Flood disasters and associated hazards in Nigeria

Adapted from Etuonovbe<sup>[5]</sup>

gion. Ezeaku<sup>[7]</sup> conducted a study on soils of selected mining areas in Nasarawa State, North Central Nigeria to assess the environmental impact of open cast mining of coal and Baryte minerals. The study revealed that the soils have been degraded, while the water resources were polluted. This is just a case study out of many mining activities going on in the country. Most of these mining activities are illegal and unethical therefore constituting great environmental hazards. Theoretically, the long-term result of environmental degradation is the inability to sustain human life. Such degradation on a global scale could imply extinction for humanity.

#### Health Risks

Most Nigerian fertilizer production is from phosphate rocks which may contain significant quantities of naturally occurring radioactive materials. At certain concentrations this becomes hazardous to health. Each year, industrial facilities discharge into the environment large amounts of chemicals leading to respiratory, neurological, developmental and reproductive disorders, and cancers. Yet, communities living within and around such industrial facilities seldom know the extent to which these discharges may be affecting their health.

## Conclusion

There is Need for fair play and sense of responsibility by the government and all stake holders in other to effectively implement EIA procures. Periodic environmental audits (EA) are recommended for all projects of possible environment impact. For the Federal Ministry of Environment to succeed in the task of environmental protection, certain basic ideas that can enlighten the people and enhance better public participation have to be conceptualized. She should develop means of making the system more indigenous without compromising the effectiveness. The agencies involved should also invest more on ground breaking scientific research that can bring innovation to our environmental protection strategies. The ultimate aim of EIA is to promote sustainable development by ensuring that development proposals do not undermine critical resource and ecological functions, lifestyle and livelihood of the communities and peoples who depend on them. Therefore, it is important to put in every effort that will enhance its effectiveness.

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The abstract presenting the purpose of the article and the main results and conclusions should contain no more than 180 words. It should be written in Slovene and English.

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A list of up to 5 key words (3 to 5) that will be useful for indexing or searching. They should be written in Slovene and English.

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Materials and methods

**Results and discussion** 

#### Conclusions

#### Acknowledgements

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[2] Higashitani, K., Iseri, H., Okuhara, K., Hatade, S. (1995): Magnetic Effects on Zeta Potential and Diffusivity of Nonmagnetic Particles. *Journal of Colloid and Interface Science*, 172, pp. 383–388.

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Seznam največ 5 ključnih besed (3–5) za pomoč pri indeksiranju ali iskanju. Ključne besede so podane v angleškem in slovenskem jeziku.

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These instructions are valid from July 2013.

Elektronski mediji morajo biti jasno označeni z imenom vsaj prvega avtorja, začetkom naslova in datumom izročitve uredništvu RMZ – M&G.

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