

Magnesia Research on Treatments for the De-Acidification of Info-Documentary Collections from Libraries, Archives and Museums: Case Studies

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The scientific concern of the authors is part of the project entitled: “The research, testing and using in the laboratory of natural products presenting significant impacts for de-acidification of paper collections”, an ambitious, original scientific project with perspective steps in ecological development and application of technological, clean lines, about the info-documents de-acidification for paper from libraries, museums and archives. Testing the natural products in pure chemical structures or composite structure (structure-blending) and obtaining of the stable chemical formula of the product is the first phase of the project which will check several products with destabilizing effects of hydrogen ions in the structure of the paper.

Keywords: *de-acidification of collections; magnesia; paper acidity; Magnesia test (magnesium oxide)*

1. General aspects. Purpose. Objectives

1.1 General aspects

The usage of new wide-spread de-acidification methods of info-documentary collections represents a very fresh scientific concern, due to the massive loss of info-documents from the great libraries, archives and museums around the world.

The irreversible effects of the acidity on the information-carrier organic supports are considered as being the most destructive and complex processes of degradation because the “paper cancer” acts in time with cumulated and irreversible effects.

The losses are immense due to the fact that there are no constant preoccupations in the great libraries regarding the monitoring of the pH evolution in the case of info-documents, by the help of the modern PH-meters, as well as of the preventive and curative interventions of mass de-acidification and the reinforcement techniques (consolidation-strengthening) of the treated info-documents.

For this reason, viable programmes appeared in the great libraries around the world regarding the salvation of the written values on organic support, the specialists in conservation receiving financial resources, high-capacity de-acidification equipment and conservation laboratories endowed with modern monitoring and intervention techniques.

The usage of the de-acidification methods, within the great libraries of the civilized world, represents the roughest preventive intervention, considered by the specialists in conservation “a necessary evil” for the neutralisation of the incipient forms and/or developed by the acidity on the information-carrier organic supports.

The safest way to slow down and block the appearance of the acidity in the info-document collections consists in: the removal of the acid processing system of the paper; the usage of the common equipment for air conditioning in museums, libraries and archives; avoidance of pollutant agents and their monitoring; the usage of some polymers for the protection of mechanical and cellulosic pastes within the process of colour reversion induced by light; avoidance of using optical bleachers when producing sustainable paper; surface treatment with starch and casein solutions; introduction of some buffering substances into the paper to decrease the influence of the oxygen in the microclimate of library, archive and museum; treatment of paper with non-volatile antiseptics for the induction of resistance to the attack of cellulolytic micro-organisms, as well as of the paper fauna; the cautious usage of ionizing radiations; the usage of modified cellulose types (with graft groups) or of the synthetic fibres when producing paper.

For the removal of the acidification sources of the info-cultural goods, due to the acid environment used in the paper producing processes or by the installation and activation of biological agents specific for the organic supports, the neutral/alkaline system with *alkyl ketene dimer (AKD)* and *alkenil succinic anhydride (ASA)* is largely used for paper sizing. The method of paper sizing and the usage of neutral-alkaline system were introduced in Romania since 1994, removing thus, the main cause regarding the acid environment in the paper producing processes, as well as in the conservation process of info-documentary collections.

For an appropriate approach of the pre-acidification, acidification and post-acidification processes of the info-documentary collections, it is necessary to present the *two systems of classification of the factors that induce the acidification of info-documentary collections*.

A first classification of causes that can cause the acidification of these goods, divides the causes into two main groups:

- *group of internal and endogenous factors* - that is specific to the paper chemistry, respectively the nature and quality of raw materials and technological conditions of production
- *group of external or exogenous factors* that defines the influence of the library, archive, museum microclimate, but also of the environment in general (Anon, 2009).

It is worth mentioning that the endogenous and exogenous factors can mutually potentiate themselves, intensifying the activation of the destructive chemical reactions; they can easily become inhibiting-blocking factors of the acidification chemical reactions of the documents on organic support by the appropriate usage of a group as blocking factor for a category of endogenous or exogenous factors.

From the presentation of the analytical classification domain of the factors that induce the acidification of collections from libraries, museums and archives, it is inferred that the essence of installation and development of the well-known rod, also named in the specialty literature as the “*paper cancer*” consists in the decryption and control of the relationships between the chemistry of organic supports that are information-carriers and the abiotic microclimatic and biotic factors.

1.2 Purpose

The aging processes of the organic information carriers, as well as those of cellulose acidification and oxidation represent processes, sometimes inevitable, of complex degradation of the document collections from libraries, museums and archives that lead in time to the change in physical and chemical characteristics, respectively to the destruction process of the cellulose.

As a consequence, the aging of the organic information carriers under the form of information products represents the whole complex of destruction of the material support and information, due to the cumulated action of endogenous and exogenous factors that cause the following reactions: reactions of oxidation with the oxygen in the microclimate of the library, archive and museum; reactions of hydrolysis under the action of relative humidity in the air; reactions of acidification of the collections in the libraries, museums and archives, due to the action of the reaction factors (pollutants, humidity) as well as of the activation factors in the case of the acidification reactions of the documents on organic support (temperature, light).

Thus, the aging process of the collections on organic supports takes place in a highly acidic environment, being a natural irreversible and unavoidable process - in time all documents being deteriorated.

An instrument that is already used for many years in the great western libraries is represented by the implementation of some programmes regarding the de-acidification treatments of the collections on organic support. There are intervention techniques, well-known in the practice of collection conservation on organic support, methods that through the test made in the research laboratories, checked the safety and efficiency of the chemical mechanisms of dislocation of the hydrogen ions from the structure of the material supports that are information carriers.

From the classical method of washing the documents with a very acid pH (<5) cumulated on a very long period of time and in precarious conservation conditions, to the modern methods by the conditioning and application of alkaline solutions under the form of spray or other modern physical-chemical methods, of accelerating the effects of chemical products used in the treatments by using the electromagnetic waves produced by a mobile device, are some of the well-known methods in the practice of applying de-acidification treatments of the collections on organic support.

The lack of national, regional and local programmes, supported by appropriate technical logistics and financed with the resources necessary to optimize the whole working process were and remain the obstacles to extensively apply the de-acidification treatments of the collections on organic support in libraries, archives and museums.

1.3 Objectives

The main objectives that have been researched in the lab and tested in the practice of the collection conservation are highlighted punctually:

- research regarding the usage of the *Magnesia* product as an efficient method in the application of the de-acidification treatments of the collections from libraries, archives and museums;
- laboratory research regarding the elaboration and application of the experimental materials and methods to test different concentrations of the *Magnesia* (2%, 1.5%, 1 %, 0.5%); case studies in laboratory conditions;
- research regarding the determination of the pH by the method of surface sampling - elaboration and application of experimental materials and methods for the determination of the paper acidity by using the pH-meter NORONIX PHT 140; drawing the evolution histograms of pH readings;

undertaking statistic calculus;

- research regarding the usage of the method by washing the acid documents in special laboratory basins by using the *Magnesia* product, finely grinded, dissolved in distilled water; establishment of the maintaining times of the documents in the reaction environment;

- research, elaboration and application of the synthesis indices used in the de-acidification treatments by washing the info-documentary collections from libraries, museums and archives;

- elaboration and presentation of the statistic analysis on the tested working variants in the experimental materials and methods;

- characterization of the washing methods in distilled water and magnesia of the highly-acidic documents, establishing the exposure time to the reaction environment; establishing the drying time of the treated documents; checking the physical, mechanical and chemical indices after treatment; conclusions; recommendations;

- research regarding the usage of electromagnetic waves in the de-acidification treatments of the collections from libraries, museums and archives treated with de-acidification products

production in the lab of the de-acidification products of the collections in libraries, museums and archives and capitalization of the products requested by the owners of cultural goods - physical and legal persons;

- elaboration of the de-acidification product technologies in safe and efficient conditions in the treatment applications.

2. Materials and Research Methods.

Elaboration and Presentation of the Experimental Materials and Methods

2.1 Materials

Magnesia product, a reagent for lab analysis, a non-harmful product according to EU legislation, was used to apply the de-acidification treatments to the info-documentary collections on paper in the laboratory.

Some specifications on magnesia, used as a de-acidification product, are presented: the product contains 99.5% magnesia; it is insoluble in the acetic acid 0.05%; it contains: chlorides 0.018%; sulphates 0.04%, heavy metals 0.003 %, arsenic 0.00004%; iron 0.069%; water solubility 0.28%. Other materials: distilled water with a pH of 6.9; special basin having a capacity of 10 l used for the preparation of the solution and application of the treatments by washing-immersion of the documents submitted to the de-acidification interventions; safety gloves, mask, and glasses; spraying device AS-2; ventilator.

The used infrastructure includes: pH-meter of Checker type, Hanna production, with electronic display for determining the pH of the reaction solution; pH-meter Noronix PHT 1140- for the acidity determinations of the documents before and after the de-acidification treatments; lens with led light - for symptomatologic observations; electronic balance Tanita, model No. 1475 - to weigh the magnesia and the preparation of the working variants; electric oven, model Memmert - for the usage of the electromagnetic waves as factor of activating the de-acidification reactions of the documents on organic support; electronic microscope, model Kruss (A Kruss Optronic) - for the microscopic analysis.

2.2 Research methods used for the determination of the pH by surface readings

2.2.1 For the determination of the pH in the case of the patrimony documents

It is recommended as the used methods to comply with the condition of maintaining the morphological integrity of the cultural good. As a consequence, the determination methods of the pH for the paper support that are classified information carriers must be non-invasive and non-destructive from a physical, mechanical and chemical point of view.

From the point of view of the investigation methods and/or non-invasive intervention, these procedures do not affect the morphological structure of the document physically, thus maintaining its integrity and the time patina (colour, glaze that is given to a cultural good with decorative purpose, to create the impression of aging or to protect it against the chemical corrosion or biological attack).

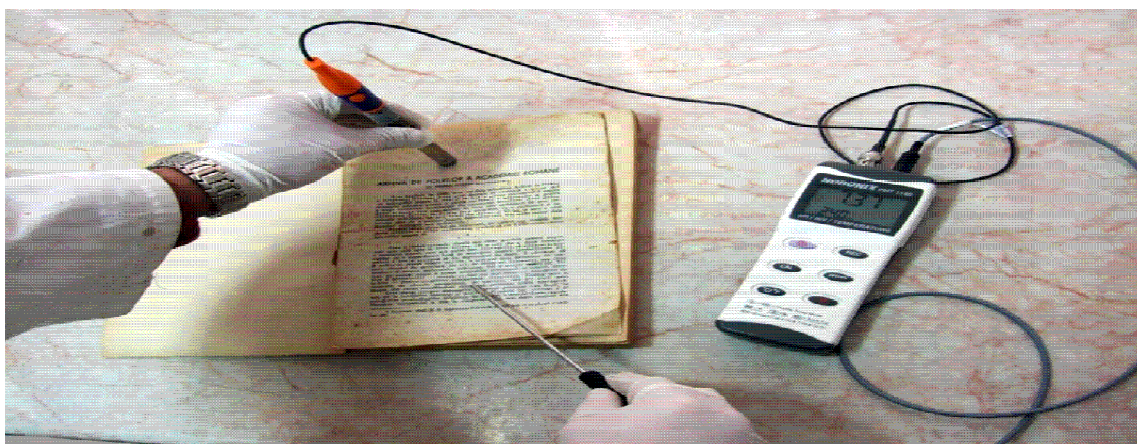


Photo 1. *Advantages of surface readings having direct contact with the paper surface that is information-carrier, by using the pH-meter Noronix PHT 1140*

- *determinations of paper acidity with this equipment represents one of the few techniques agreed in the monitoring, storage and interpretation of the acidity for the info-documentary collections from libraries, museums and archives;*
- *the surface readings with this equipment are executed with a pH - meter with electrodes adapted technically to operate in a liquid environment;*
- *by placing the electrodes in contact with a drop of distilled water on the paper surface, an aqueous mini environment is formed wherein the electrodes act through direct contact;*
- *by using this modern equipment, the measurement device of the hydrogen ion concentration in the paper can be logged into a PC to capture on-line data regarding the values of pH/mV;*
- *the records of pH/mV or temperature can be saved in documentary files and the data can be used in different analyses: operational analyses; the evolution statistics of pH readings and temperature; display of more documents on the monitor functions adapted by choice; drawing the histograms (diagrams) of pH and temperature;*
- *the keying of the portable station with the contact electrode in solution with a standard pH (pH 4, Ph 7, pH 10), by accessing the commands in an integrated electronic system removes the measurement errors up to the standard values agreed by the institutions authorised in the standardization of the measurement operations of the acidity for the information-carrier organic supports;*
- *pH-meter Noronix PHT 1140 is foreseen with the following endowments: mobile station with contact electrode for the measurement of the temperature (°C/°F); software RS232 for the storage and processing of on-line data.*



Photo 2. *Determination of pH in liquid solutions - elaboration of working methodology on the use of Hanna pH meter with electronic display*

The acidity determinations of the solution before and after the documents' washing in liquid reaction environments were made with the pH-meter Hanna, with electronic display. The first step consists in the removal of the safety helmet of the station and the attachment of the electrode to the measurement device.

Furthermore, the rod of the electrode is introduced in the liquid sample (distilled water with pH 7, before and after the immersion washing of the documents) prepared in a laboratory test tube. The content of the test tube is slightly shaken. The electronic display system is opened and the display equalization is waited for, after which the pH indicated on the monitor is read.

If a different test is needed, the washing (rinsing) of the electrode in distilled water is

2.3 Elaboration and presentation of the experimental materials and methods for making the research regarding the usage of the magnesia in the de-acidification treatments of the collections from libraries, archives and museums

From a scientific point of view, the presentation and application of the scientific methods and materials represent essential stages for the whole working process through which the scientific research instruments are established, adapted to the objectives had in view for the usage of the experiences and experimental variants for the acquisition of the scientific data; determination of synthesis indices used in the scientific process; storage and processing of data; elaboration of scientific programmes; elaboration of synthesis, conclusions and recommendations (*Table 1*).

Crt. no.	Experiment specification	Research variants	Average values of pH	Number of repetitions - number of readings pH	Obs.
1.	<i>Exp.1. Research regarding the usage of magnesium oxide 2% in the de-acidification treatments of info-documents</i>	V1. Control sample. Average pH value in the untreated document	4.4	502	
		V2. Average pH value in the document treated with magnesia 2%	7.7	502	
2.	<i>Exp.2. Research regarding the usage of magnesia 1.5% in the de-acidification treatments of the info-documents</i>	V1. Control sample. Average pH value in the untreated document	5.2	504	
		V2. Average pH value in the document treated with magnesia 1.5%	8.1	502	
3.	<i>Exp.3. Research regarding the usage of magnesia 1% in the de-acidification treatment of the info-documents</i>	V1. Control sample. Average pH value in the untreated document	5.2	502	
		V2. Average pH value in the document treated with magnesia 1%	8.5	502	
4.	<i>Exp.4. Research regarding the usage of magnesia 0.5% in the de-acidification treatments of the info-documents</i>	V1. Control sample. Average pH value in the untreated document	5.5	503	
		V2. Average pH value in the document treated with magnesia 0.5%	7.5	503	

Table 1. *The research methods and materials designed and applied for the accomplishment of research regarding the usage of magnesia in the de-acidification treatments of collections from libraries, archives and museums. “Gheorghe Șincai” County Library, Oradea, Bihor, Laboratory of conservation-research-2014 (original)*

2.3.1 Elaboration and presentation of the research methods and materials regarding the determination of the synthesis indices used in the application of the de-acidification treatments by washing the collections from libraries, archives and museums; specifications regarding the determination of indices

The elaborated synthesis indices represent the useful analytical and scientific instruments, especially in the research domains within which fields and sub-fields represented through the number and diversity of variants and working repetitions.

In the case of the research undertaken in the Laboratory of conservation-research of the “Gheorghe Șincai”, County Library, Oradea, Bihor, the application of the complex experimental materials and methods through the number and essence of variants and used working repetitions, represented a challenge for the storage, processing, interpretation and capitalization of the obtained scientific results.

The presence of the synthesis indices used as viable instruments in our scientific working process was necessary, so that the experimental results obtained on variants and working repetitions to be quantified.

Some technical calculus aspects for the main identified and analyzed indices in Table 2 have been highlighted, each index used in the de-acidification treatments of the collections being presented into details.

- a. *Index of neutralisation (IN) (pH units)* represents the difference between the pH obtained by applying the treatment and the pH of the untreated document;
- b. *Initial weight of the untreated documents (G.i.d.)*, as well as the *final weight of the treated document (G.f.d.)* are indices that are obtained by weighing the document before and after the application of the treatment;
- c. *Content in solution of the treated documents (CS) (g/document)* represents the difference between the final weight of the treated document at a certain environmental temperature and at a certain time range that is rigorously established and the initial weight of the untreated document (see Table 2);
- d. *Some useful observations are made for the paper absorption index (IPA - %/document)* regarding its determination by using as calculus structure the indices in Table 2.

The calculus formula is:

$$IPA = \frac{100 \times CS}{100 - CS}, \text{ where CS represents the content in solution of the treated document}$$

By replacing it with the value in Table 2, it is achieved:

$$IPA = \frac{100 \times 3}{100 - 3} = \frac{300}{97} = 3,09\%$$

- e. *Total capacity of absorption (CTA - g/document)* is obtained by multiplying the solution content of the treated documents with the initial weight of the untreated document

In a first phase of research, these indices are used for all the preventive humid treatments. They represent a major subject for the domain research, being convinced by the usefulness of the further working processes undertaken by the Laboratory of conservation-research from “Gheorghe Șincai” County Library, Oradea.

Crt. no.	Product used and concentration	Research variants		Neutralisation index (IN) (pH units)	Initial weight of the document G.i.d. (g)	Final weight of the treated document G.f.d. (g)	Solution content CS (g/doc.)	Paper absorption index IPA (%/doc)	Total capacity of absorption (g/doc.)	Obs.
		pH V ₁ untreated - control- (pH units)	pH V ₂ treated (pH units)							
1	2	3	4	5(4-3)	6	7	8(7-6)	9	10 (8×6)	
1.	Magnesia 2%	4.4	7.7	3.3	166	169	3.0	3.09	512.94	
2.	Magnesia 1.5%	5.2	8.1	2.9	646	648	2.0	2.04	1 317.84	
3.	Magnesia 1%	5.2	8.5	3.3	133	135	2.0	2.04	266	
4.	Magnesia 0.5%	5.5	7.5	2.0	174	176	2.0	2.04	348	

Table 2. *Research procedure elaborated and applied for the determination of the synthesis indices used in the application of de-acidification treatments for the info-documentary collections in libraries, archives and museums (according to V.A. Deac, 2014)*

3. Scientific results. Discussion

3.1 Specifications on the usage of the magnesia prepared and used as analysis reagent

The magnesia (MgO) is a formless, white powder with a slightly alkaline taste, with features useful in the protection of info-documents on organic support in the libraries, archives and museums, being an efficient and promising absorbent and anti-acid protector, with some weak anaesthetic, surface effects, that disturbs the development of the biological cycle in the case of fungi and cellulolytic bacteria, as well as of the paper and xylophages insects.

The test made in the Laboratory of conservation-research from “Gheorghe Șincai County Library, Oradea, Bihor, on lab samples from suspensions that contain colloidal bentonite and magnesia highlighted the beneficial anti-acid effects on information-carrier organic supports that were strongly acidified in time.

Other set of laboratory studies, undertaken before our final working process, highlighted the neutralising capacity of the paper acidity, quality tested in the laboratory by washing the strongly acid documents in solution made up of distilled water and magnesia prepared and used as analysis reagent.

The determinations made in the laboratory on the usage of magnesia prepared as analysis reagent present a special importance in the elaboration and application of de-acidification treatments in the case of info-documentary collections from libraries, archives and museums for the following reasons:

- magnesia prepared and used as analysis reagent is soluble in water and acids, having a strong neutralising and absorbing mechanism;
- this product can increase the pH of the info-documents in the libraries, archives and museums up to 9 units pH;
- magnesia presents anti-acid features proved in the laboratory having a significant capacity of neutralisation of the hyper-acidity in the case of the info-documents that are highly-affected by the exogenous and endogenous abiotic and/or biotic disturbances;
- treatments of the very acidic documents, by their washing through immersion in an environment made up of magnesia, neutralises the acidity of the treated documents with at least 3-5 units pH;
- the laboratory tests proved that the buffering agent is uniformly distributed on the morphological components of the treated document by washing, through its controlled immersion in the solution rigorously prepared;
- the product doesn't affect the integrity of the inks and doesn't degrade the adhesives and other components of the books or their bindings, as long as the immersion of the documents in the de-acidification solution is controlled to avoid some accidents regarding the morphological and information stability of the treated documents;
- by the usage of the magnesia under the form of analysis reagent, the finally obtained pH can range from 7 to 10 normal values and is usually located on the range 8-9.5; On this range of values, there are no risks of alkaline hydrolysis, because the maximum is ensured close to the risk limit for the initiation of the alkaline hydrolysis;
- the product tested in the Laboratory of conservation-research from “Gheorghe Șincai” County Library, Oradea, Bihor can be used without any reserve in the treatments on the de-acidification of the info-documents from libraries, archives and museums (books, manuscripts, archive registers, press, maps, documentary archive collections, other collections on paper support);

- after the application of this product in the laboratory, by using the controlled washing-immersion of the documents that are highly acidic, the mechanical resistance indices of the paper are not affected, there are no ridges or other deformations of the paper, but there is recorded a slowing down process of the degradation mechanisms by the control and blocking the chemical degradation;
- the magnesia product under the form of analysis reagent but also the reaction solution are not toxic for the health of the operators; they do not contain CFC and solvents;
- for the perspective research of the Laboratory of conservation-research from “Gheorghe Șincai” County Library, Oradea, Bihor, some modern solutions of processing the alkaline buffering product will be found to neutralize the paper acidity.

3.2 Experimental procedure

For the dissemination of the obtained scientific results it is necessary to present the experimental procedure used in the application of the neutralising treatments of the hyperacidity of the documents on organic support, with special references for two analytical structures: the presentation of the general experimental procedure that includes the general elements with macrostructural implications; presentations of the specific experimental procedure with special references on the variants, sub-variants and number of repetitions used for data sampling, used experimental technique and data sampling, storage and processing methodologies. Within the developed research, the experimental procedure represents the pattern necessary for the scientific process that coordinates all the activities made on experiences, variants and working repetitions.

This procedure is necessary to be presented into details for the multiplication of the research in other conditions of library, museum and archive according to a minimum of technical equipment that is necessary to make the treatments.

The experimental procedure will be considered a scientific one when the scientific results will be presented and the calculus methods will be applied by statistic techniques in order to establish efficient working variants in the application of the treatments for correcting the paper acidity.

3.3 Summary of the scientific results obtained on the elaborated and applied research procedure

The scientific results on the application of the magnesia in different concentrations were highlighted in Table 3.

Out of this table analysis, some precise and useful information can be obtained in the practice of library collections' conservation, representing a starting point in the new approaches on the de-acidification of library collection by using nano-particles of magnesia. The research designed and undertaken in Oradea are part of an ample research programme that has in view useful and affordable technical solutions for the practitioners.

Crt. no.	Research variant	pH of an untreated document - control sample				Ph of a treated document			
		Maximum value	Minimum value	Average value	Std. dev*	Maximum value	Minimum value	Average value	Std. dev
1.	Magnesia 2%	6.0	3.9	4.4	0.4	8.2	6.9	7.7	0.4
2.	Magnesia 1.5%	6.1	4.7	5.2	0.4	8.6	7.8	8.1	0.1
3.	Magnesia 1.0%	5.9	4.8	5.2	0.3	8.9	8.4	8.5	0.1
4.	Magnesia 0.5%	6.0	5.3	5.5	0.2	8.1	7.1	7.5	0.3

* standard deviation of the pH-meter (std.dev) highlighted by the statistic calculus

Table 3. Summary regarding the synthesis of the research undertaken for the usage of the magnesia (2%;1.5%; 1.0%;0.5%) in the de-acidification treatments of the collections from libraries, museums and archives (“Gheorghe Șincai” County Library Bihor, Oradea - Laboratory of conservation-research 2013-2015)

The laboratory research made at “Gheorghe Șincai” County Library, Oradea proved that the magnesia product in the researched concentrations represents an efficient solution for the neutralisation acidity of the paper for all the library documents, as it follows:

- bound documents (books, magazines) can be treated by washing them with solution page after page and in the case of very high acidic values (under 5), the immersion in special basins is recommended;
- for press and other documents that are not bound, the treatments with this product can be made by applying a spray solution;
- for the interventions made on the patrimony documents, the laboratory tests made in Oradea with this product highlighted its efficiency in neutralising the acidity spots under the form of individual or united glomeruli (aggregates of variable sizes, formed by the agglutination of hydrogen particles from the cellulosolytic structure of paper).

The paper acidity spots as well as the action of the magnesia have been highlighted in the photos below.



Photo 3. Paper acidity spot under the lens of the laboratory researchers

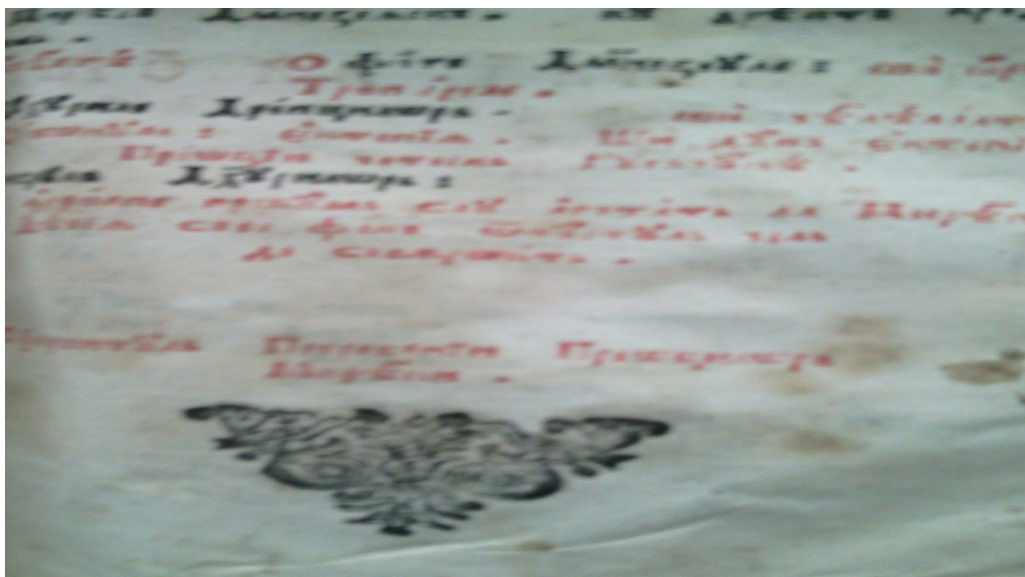
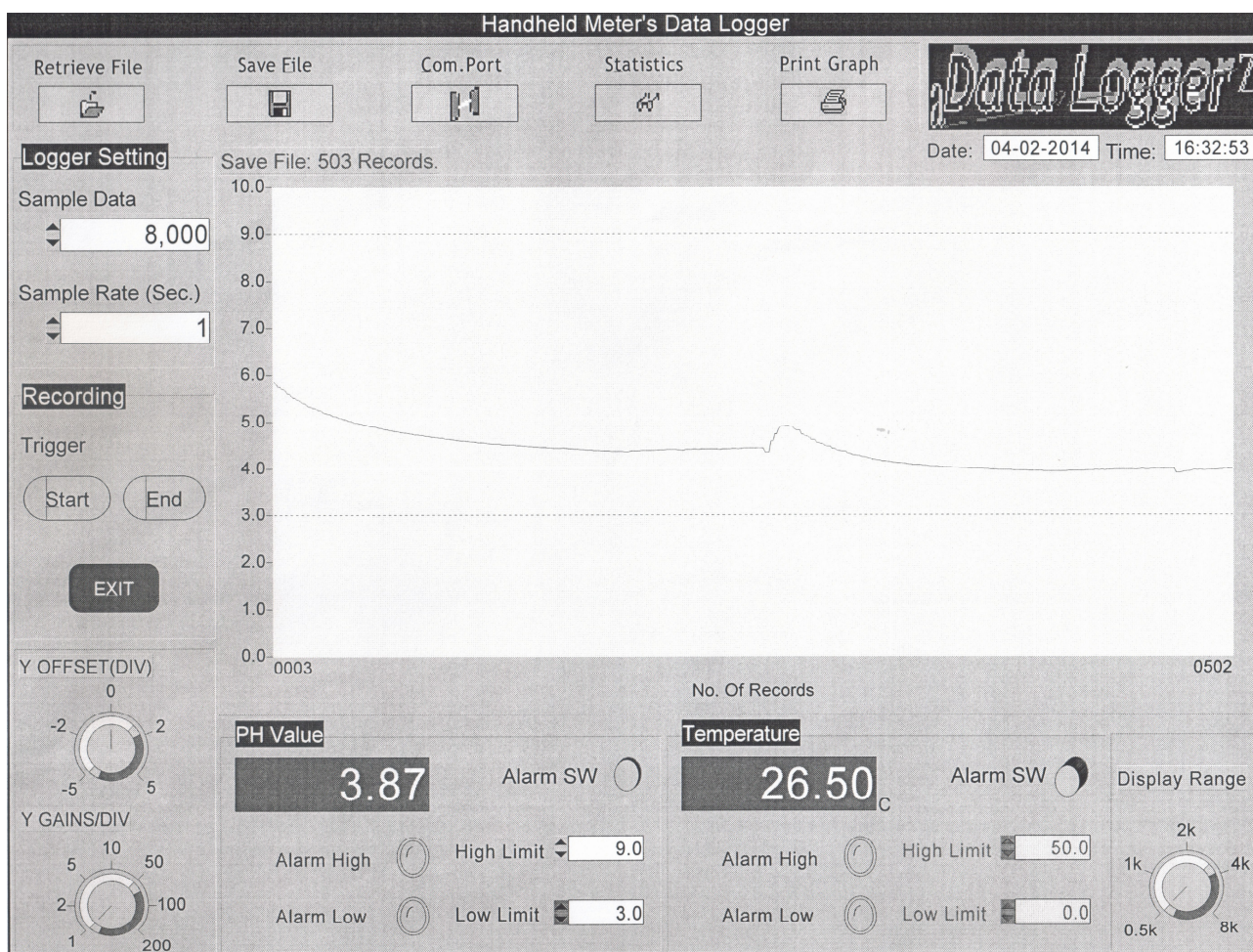


Photo 4. Final effect of the magnesia in neutralizing the acidity spots

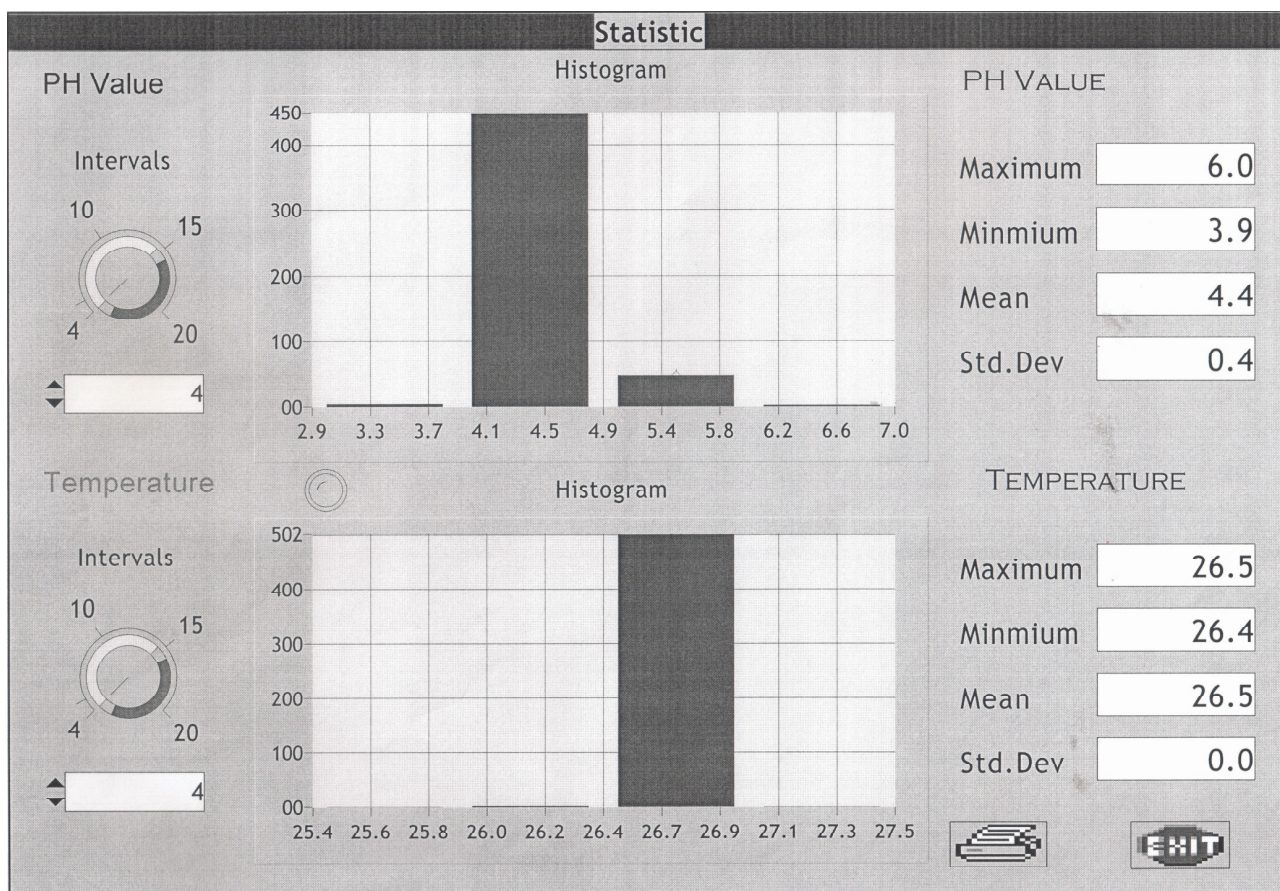
ANNEX 1

Determination of paper acidity by using pH-meter NORONIX for control variant (before the treatment with magnesia 2%)



ANNEX 2

Statistic calculus for the researched variant: presentation of histogram, as well as of maximum, minimum and average values of the pH for control variant (before the treatment with magnesia 2%)



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