Funori seaweed extracts as alternative material to semi-synthetic products used in paper conservation

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Abstract

Green approach to conservation is becoming increasingly popular in the recent years, focusing on finding products that could provide a guarantee of stability over time, with a view to perform reversible, compatible and less invasive interventions on the object. The natural adhesive and consolidant algae extract, commonly known as *Funori*, were introduced and tested for the resizing of paper materials. In this paper are discussed the results of several tests performed to analyse Funori seaweed extracts within restoration of paper as a green solution.

Keywords

Funori, Seaweed extracts, Paper conservation, Green conservation, Resizing

Introduction

From the moment of its invention and diffusion, paper has replaced other supports as material mostly used by mankind to transmit its own testimonies. Like all organic materials, paper is destined to deteriorate over time and, to avoid this loss, it is necessary to plan projects that prevent degradation and at the same time secure those works of art already damaged. It is therefore necessary to find methods, to study products and treatments that can forecast or, at best, slow down the natural degradation of paper (Lalli, Kron-Morelli, Brogi, Baudone, & Tosini, 2016; Baudone, 2019a, 2019b).

For our research, we focus on resizing, the intervention indispensable to reintroduce the correct quantity of glue inside the meshes of paper fibres, that was naturally lost due to natural ageing as well as previous conservative operations. The presence of glue is essential to paper, since it allows it to have those characteristics of writability, resistance and protection against atmospheric agents, wear and the natural degradation of time. The internal structure of a paper sheet is a confused overlap and interweaving of fibres on each other and the final product is highly absorbent, unsuitable for the purposes. The operation of sizing is necessary, that is to add adhesive substances that regulate the absorption of liquids while giving the paper certain physical and mechanical properties (Roberts & Etherington, 1981). Therefore, the importance of resizing becomes evident during the conservative treatments that accompany the restoration of a paper object. Obviously, this operation is strictly bound to the type of support to treat, to its solubility problems and to the final aim of the intervention to be performed.

In addition, the materials used in resizing operations are various, consisting mainly in cellulose ethers, prepared from aqueous or non-aqueous solutions, depending on the type of object to be handled, and the consequent reactions evoked on the paper surface following graphic medium.

Percentages also change, depending on the type of material to be treated, according to its thickness, flexibility, rigidity, porosity and graphic mediations present. It is a common and widespread opinion, in the field of paper conservation, that a suitable percentage of the correct amount of adhesive for the resizing of paper support is included between 1 or 2%, regardless of the nature of the adhesive used as well as the nature of the raw material constituting the artefact itself (de Bella *et al.*, 2005). Each kind of paper is an independent support, with its own physical and chemical properties, with its degradation processes and it should require a peculiar procedure of conservative intervention.

The introduction into the paper fibres of an excessive quantity of adhesive can have extremely counterproductive effects on the future conservation of the object, such as the appearance of surface tensions due to a non-homogeneous absorption of the adhesive by the paper, in particular if ancient and handmade, where each *bifolium* is different from the others; loss of elasticity and flexibility due to the excessive accumulation of glue on the surface, creating a layer that alters the natural mechanical properties of the paper; difficulty in react to the surrounding environment, slowing down or complicating the achieve for a thermo-hygrometric equilibrium due to the saturation of the pores present between the fibres. Percentages lower than 1% have a high penetration capacity inside the paper fibres, conveying the correct and sufficient quantity of adhesive and preventing the formation of thick adhesive surface layers, due to the presence of an excessive quantity of adhesive. Keeping these elements in mind, we tried to find an alternative to the common cellulose derivates and adhesives used in resizing paper materials. The seaweed extract commonly known as *Funori* seems to respond very well to all of the requirements needed for this aim: excellent cohesive and adhesive power even at low percentages; low viscosity degree; neutral pH with slightly tendency to alkalinity; complete affinity with aqueous solutions and total reversibility. In addition, this study tested the ability of *funorans* to withstand the development of microorganism, slowing or inhibiting their growth. The achievement obtained during these experiments gave us very encouraging results (Lalli, Kron-Morelli, Brogi, Baudone, & Tosini, 2016; Baudone, 2019a, 2019b).

Materials and Methods

Funori is the generic name given to the algae species of the genus *Gloiopeltis*, mentioned for the first time in Japan, as a sizing agent for textile and paper materials, as an additive and thickening agent for mortars, plasters and ceramic products, in the areas of food, cosmetics and pharmaceutical productions. The algae species, which the adhesive is extracted from, generally known as *funorans*, are substantially three and they all are much analogous to each other: *Gloiopeltis tenax*, *Gloiopeltis furcata* and *Gloiopeltis complanata* (Chapman, 1970). This extract is a lightweight adhesive, which allows for its use in controlled operations, even when carrying out several substance applications in sequence. It is considered a good surfactant, capable of increasing water wettability and is often used together with wheat starch paste or animal glue, to lower the viscosity of the solutions. *Funori* shows good optical properties (Webber, 2006; Geiger & Michel, 2005; Michel, Geiger, Reichlin, & Teoh-Sapkota, 2012) and a substantially neutral pH, variable according to the solution. The final pH of the solutions depends by the seaweed itself, the purity of the product, the method of extraction and the water used.

In the last fifteen years, research and studies regarding this product have recorded a considerable increase, although to this day, *Funori* still remains scarcely widespread. However, this is a sufficient stimulus to deepen the study of a product that could be a valid alternative to the most recent semi-synthesis products used in the paper materials conservation. Traditionally, in Japan a mixture of three algae extract is used: *Ma-funori* (*Ma* - true, from *Gloiopeltis tenax*), *Fukuro-funori* (Fukuro - balloon, from *Gloiopeltis furcata*) and *Hana-funori* (*Hana* - flower, from *Gloiopeltis complanata*), which differ from each other in some properties concerning primarily the adhesive power, the viscosity and the solubility in water. These differences are also linked to the method of extraction, the place of origin and the mixing percentages of these three types of seaweed. The *Ma*- has a more effective adhesive function, the Fukuro- has a less adhesive power but is easily soluble in water, while the *Hana*- doesn't show the same adhesive properties as both other ones. Therefore, is generally used as a colloidal substance, since it allows the extracted materials to remain dispersed in the aqueous solution (Hayakawa, Araki, Kainuma, Taguro, & Kawanobe, 2004). Moreover, it is a highly water-soluble substance due to the short polymeric chain, simultaneously showing a good resistance to the humid environment, in addition to a certain chemical stability over time.

Funori is a very complex polysaccharide, with great affinity to cellulosic materials. It is a natural polymer whose chemical structure can show different forms that characterise its adhesiveness, viscosity and solubility in water, placing itself halfway between the agar and the carrageenans. *Funori* has the same basic structure as agarose (β-D-galactose + 3,6-anhydro-a-L-galactose) but is sulphated, in a smaller percentage, like carrageenans (β-D-galactose + 3,6-anhydroa-L-galactose + sulphate). Theoretically *Funori* could be considered an galactose-6-sulfonate, (β-D-galactose-6-sulphate + 3,6-anhydro-a-L-galactose) a sort of fractioned agar containing sulphate elements (Tuvikene *et al.*, 2015; Geiger & Michel, 2005; Takano, Hayashi, Hara, & Hirase, 1995; Hirase & Watanabe, 1971; Hirase, Araki, & Itō, 1956). Its antimicrobial activity is important (Ren, Noda, Amano, & Nisikawa, 1994; Zheng, Chen, Yao, Chen, & Shi, 2012), capable of slowing down or altogether inhibiting the development of microorganisms that are harmful to the integrity of the works of art, as well as its property of not altering the optical refraction index of the surfaces, which it is spread on.

Studies previously conducted on *Funori* highlight a certain plurality of preparatory procedures (Swider & Smith, 2005; Evans, 1984; Masuda, 1984; Masako, 1979), all of them enough similar to each other. We remember that percentages, cooking times and dilution factors, source of purchase or simply the various preparatory phases are not whenever reported. The fact that *Funori* is rarely manipulated before being put on the market can be encouraging about the purity of the material to be used on works of art, although, depending on the supplier and the methods of cultivations, there may be chemical treatments of the seaweed, after rinsed and dried in the sun with sodium peroxide or natrium hydroxide solution involved in the process.

Distributed in various forms, from dried algae sheets to powder, up to the recent liquid form, *Funori* solution is used in conservation as an aqueous adhesive for veiling and pre-consolidation operations of paper supports fixed on canvas in view of their detachment (Landwehr, Schuster, & Zeilinger, 2011; Polidori, 2010); as a consolidator at various percentages for pictorial mediums on various types of paper (Harrold & Wyszomirska-Noga, 2015; Gregory, 2012; Gohde Sandbakken & Storevik Tveit, 2012; Catcher & Burgio, 2005; Madani, 2004), silk (Blair & Thompson, 2014; Gao & Hsiao, 2013), papyrus (Menei & Caylux, 2013), wood (Thuer, 2011) paintings (Geiger & Michel, 2005; Carnazza & Corak-Rinesi, 2005) and wall paintings (Catenazzi, 2017; Lalli, Kron-Morelli, Brogi, Baudone, & Tosini, 2016);combined with other adhesives, such as isinglass or wheat starch paste, to improve lowering of viscosity of the solutions (Daniels, Hacke, Qui, & Marabini, 2013; Gregory, 2012; Belard, 2010; Prêvôt & Sindaco, 2008).

As can be seen, the treatments of conservation and restoration involved in these studies and applications are as various as methodologies. In all cases, its qualities of mild adhesive and surfactant are exploited, capable of dissolving in water at room temperature and penetrating inside the supports, which it is laid on.

Generally, a solution can be prepared in two ways: hot or at room temperature. In literature, speaking of the physical properties of this product, it almost always means that the solution is prepared hot, in the traditional way. The studies about the results of a preparation of *Funori* solution at room temperature are scarce. To overcome this issue and trying to open a new debate, tests of this study were performed with a solution prepared at room temperature, using dried seaweed sheets (Lalli, Kron-Morelli, Brogi, Baudone, & Tosini, 2016, 2019b).

For our study, natural dried algae sheet were used, purchased directly from Mie Prefecture, Japan. Differences between both procedures essentially concern the density of the solution, its adhesive power, the ability to penetrate into the fibres and the final appearance. The concentration used was established by referring to the standard percentages used for cellulose derivatives in resizing operations, in order to obtain comparable results. Furthermore, the choice to prepare the solutions at room temperature is related to the same methodology used for the preparation of cellulose adhesives, making it therefore possible to compare the density and viscosity of both products. The choice of room temperature preparation has another motivation, too towards the production of an easily penetrable adhesive, with very low viscosity values and weak adhesive power, necessary and sufficient to keep the fibres cohesive inside the paper support without altering their physical and mechanical properties. The dried seaweed is previously rinsed in water and then put to swell in a given amount of water for a time ranging from six to twelve hours – the time is relative to the percentage prepared and to its intended use. Later, it is filtered through a fabric, most often cotton (Lalli, Kron-Morelli, Brogi, Baudone, & Tosini, 2016; Baudone, 2019a, 2019b).

Each percentage of Funori prepared was subjected to pH measurements, using Hanna Instruments® HI-99171® portable pH meter supplied with HI-1414D® flat tipped pH/temperature probe: all readings, taken six times per sample, gave us an average value that is included within the neutral range with a slight trend to basicity - an optimum assumption for an adhesive to be used on a material that is extremely sensitive to acidic and alkaline environments, such as a paper material (Table 1). A high level of acidity, as well as a high accentuated presence of alkalis, can trigger chemical reactions of hydrolysis and oxidation that would affect the degradative processes of the artwork, worsening the conservative state and putting at risk the usability of the object.

Percentages %	Funori **(μ)**	Tylose® MH 300p **(μ)**	Glutofix® 600 **(μ)**
0.3	7.36	7.04	7.14
0.5	7.29	7.08	7,17
0.7	7.37	7.09	7,16
1	7.45	7.14	7,23
1.5	7.38	7.18	7,25
2	7.41	7.21	7,29

Table 1. pH average measurements of *Funori* solution in comparison to Tylose® MH 300p and Glutofix® 600 prepared at room temperature pH average deionised water: 6.89

The characteristics of an adhesive are also determined by its tendency to be susceptible or not to the proliferation of biological agents. These elements participate in the degradation process of the artefact, with damages that are unrecoverable on paper. There are a lot of microorganisms that attack paper materials, attracted by the cellulose itself or by those substances present on its surface, such as adhesives. The alteration of the starchy and proteinaceous glues due to biological attack is well documented (Skeist, 1997). Although the *Funori* solution extracted from *Gloiopeltis* is of organic origin, numerous researches (Zheng et al., 2012; Inagaki, Saeki, & Ishihara, 2011; Fang et al., 2010; Kurihara, Goto, Aida, Hosokawa, & Takahasi, 1999) ensure the ability of *funorans* to withstand the development of microorganisms, slowing or altogether inhibiting their growth. Upon completion of the research, it was indispensable to perform vitality tests to corroborate or refuse these statements, as well as to have at the same time a complete picture of the performance of

Funori employed in the field of paper materials. Two different viability tests were carried out, using Petri dishes with agar media and microscope slide holder without media, to observe the development of microorganisms in environments that had or had not a nutritional basis (Lalli, Kron-Morelli, Brogi, Baudone, & Tosini, 2016; Baudone, 2019a, 2019b). A small and identical quantity of each adhesive (modified wheat starch paste, animal hide glue, Vinavil® 59, Glutofix® 600, Tylose® MH300p, Klucel G®, *Funori* solutions 0.3%, 0.5%, 0.7% and 1%) was placed inside the Petri dishes, while on the slides each adhesive was applied in five consecutive layers. There was no inoculation of microorganisms in any of the Petri dishes or on the microscope slide holder: the samples were left open, exposed to the air for a couple of hours and then closed inside a plexiglass box with conditions that reproduced extremely favourable environmental conditions for the microorganism growth (Lalli, Kron-Morelli, Brogi, Baudone, & Tosini, 2016).

After these preliminary tests, resizing experiments were carried out using *Funori* seaweed extracts on different types of both hand-made and machine-made paper supports. Similarly, the graphic mediations present on paper supports differed from each other, ranging from manuscript iron-gall ink to modern Indian ink, up to ancient and modern printing inks. Solubility and pH measurements, optical microscope observations, wettability test estimating the time a drop of water needed to be absorbed, histochemical staining tests to search the original adhesives and other products used during papermaking processes were carried out on each specimen, in order to obtain a framework including all the main aspects concerning the paper samples to be processed.

All the paper samples were mechanically dry cleaned; tested the solubility of the inks with hydroalcoholic solutions at various percentages and measured pH value; washed by immersion in deionised water and left dry at room temperature upon frames, measured again the pH of the washed paper; resized with *Funori* solutions at various percentages and left to dry again on frames, subsequently completing the drying operations under light weight; further pH measurement for a comparison value with the initial reading and finally the conservation of the various samples in environments with different level of Relative Humidity (RH) and temperature, in order to observe the behaviour of the adhesive in various environmental conditions. This selection was suggested by the desire to observe the response of a resized paper with *Funori*, knowing its chemical and physical nature.

To conclude this study and to observe the behaviour of both paper samples and *Funori* solution over time, each test performed was subjected to monitoring in environments with different temperatures and relative humidity percentages, in order to determine whether the adhesive could lead to changes or variations such as stiffening, yellowing or browning, increased acidity or alkalinity, analysing the physical properties and measuring the pH at regular intervals of three months. Environmental conditions that affect the object, affect also all individual elements, the object is composed of. Even the adhesive substances are involved in the search for equilibrium between the object and its surrounding environment, playing an important role in the protection of the work of art - or in the acceleration of its degradation. Each adhesive substance has its own particularities that make it react and behave differently in the various types of environments. In order to observe the environmental adaptation of a resized paper with *Funori*, the various specimens used for the experimentation were conditioned in different conservative environments, carrying out a quarterly rotation of the environments with pH measurements for each paper support studied. All papers tested in this study were located inside a folder made of thin cardboard sheet with neutral pH and the whole package wrapped in paper: these measures were used to keep the papers in an acid- or alkali-free environment, in order to avoid an unnatural alteration of pH values with consequent inaccurate readings. The principles for the execution of this monitoring were chosen based on two very important elements in the field of conservation: temperature and relative humidity, although the factors to be considered are more and include light, mould, insects and other pests, air and dust pollution. In this case, placing the samples between a folder and wrapping the whole package, the problems caused by light, mould, insects and various pollution do not arise. For both factors, four different combinations have been found, which can ensure the change of at least one of both components. Samples were stored separately, grouping them according to type and avoiding putting them directly into contact with each other.

Results and Discussions

In the preliminary tests, *Funori* seaweed extracts showed their resistance to the proliferation of microorganisms, meanwhile the other adhesives showed a various proliferation of biological elements.

Although microorganism growth evaluation needs specific protocols and so these results must be evaluated cautiously. We can expect for a similar result even in the conditions of the conservative standards indicated for paper materials. However, a generalisation appears out of place, given that the combinations of elements that can lead to the proliferation of biological agents are multiple and not linked solely to the conservative environment, but also to the nature of the material itself and its usability.

Regarding the resizing tests, the percentages of *Funori* included between 0.3% and 0.7% gave us the best results for all types of papers tested (Table 2), a result very similar to those obtained with 1% Tylose® MH 300p. The adhesive has perfectly penetrated inside the meshes of the paper supports, giving them the suitable mechanical consistency without altering their flexibility, elasticity and, at the same time, giving the paper a natural and uniform appearance. In addition, other important factors have been noted: on the paper surface there were no halos or lucidity of any kind, showing that a dried film application of *Funori* seaweed doesn't alter the paper surface on which it is laid; moreover, as can be well noted, all the pH average values fall within a slightly alkaline pH, within that range of values capable of buffering the chemical reactions of hydrolysis and oxidation. A preliminary conclusion about the results of these tests pushed us towards encouraging considerations. *Gloiopeltis* extract would appear to be a more than valid alternative to the common cellulose derivates, capable of transmitting the correct amount of adhesive needed and sufficient for the paper support at the correct concentrations to reacquire its natural chemical and physical properties.

Samples	pH value unwashed paper (μ)	Initial thickness (mm)	pH value washed paper (μ)	Resizing solution % *Funori*	Resizing solution % Tylose® MH 300p	pH value after resizing (μ)	Thickness after resizing (mm)
B_1	6,77	0,083-0,106	7,11		**1**	7,74 Δ[+0,63]	0,083-0,106
B_2	6,79	0,075-0,098	7,24	**0,3**		7,82 Δ[+0,58]	0,075-0,098
B_3	6,69	0,084-0,104	7,18	**0,5**		7,77 Δ[+0,59]	0,084-0,104
B_4	6,81	0,083-0,110	7,29	**0,7**		7,86 Δ[+0,57]	0,083-0,110
B_5	6,66	0,091-0,113	7,12	1		7,75 Δ[+0,63]	0,091-0,113
B_6	6,71	0,077-0,088	7,16	1,5		7,81 Δ[+0,65]	0,077-0,088
B_7	6,74	0,069-0,086	7,10	2		7,73 Δ[+0,63]	0,069-0,086

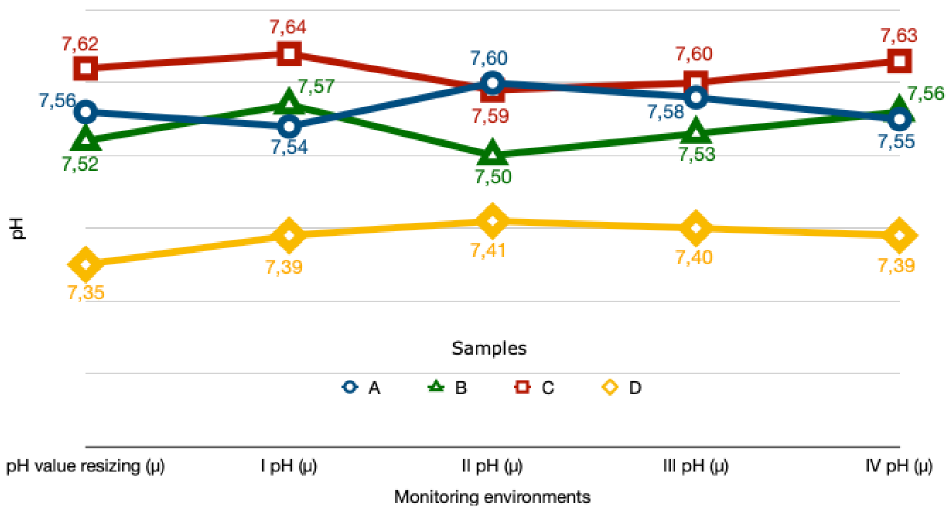
Table 2. Test results on sample B, ancient handmade paper with printed ink

After one year of monitoring in environments with different temperature and RH percentages (Table 3, Graphic 1), none of values recorded on the samples tested with *Funori* solution underwent drastic variations. We have also found that *Funori*, like the most commonly used cellulose ethers, manages to maintain its physical and chemical properties when environmental conditions of temperature and relative humidity change.

			pH average values (μ) of the first environmental monitoring cycle			
Samples	*Funori* solution %	pH value after resizing (μ)	I 10°C + 40% RH Ph ±	II 25°C + 40% RH Ph ±	III 10°C + 60% RH Ph ±	IV 25°C + 80% RH Ph ±
A	0,3	7,56	7,54 Δ[-0,02]	7,60 Δ[+0,04]	7,58 Δ[+0,02]	7,55 Δ[-0,01]
B	0,3	7,52	7,57 Δ[+0,05]	7,50 Δ[-0,02]	7,53 Δ[+0,01]	7,56 Δ[+0,04]
C	0,3	7,62	7,64 Δ[+0,02]	7,59 Δ[-0,03]	7,60 Δ[-0,02]	7,63 Δ[+0,01]
D	0,3	7,35	7,39 Δ[+0,04]	7,41 Δ[+0,06]	7,40 **Δ[+0,05]**	7,39 Δ[+0,04]

Table 3. pH average measurements of handwritten supports on handmade paper with iron-gall inks (A-B-C) and machine-made paper with Indian-Ink (D) after one year of environmental monitoring

At the same time, at a first tactile observation, all the papers maintain their mechanical properties of elasticity and flexibility, without altering the naturalness of the sound during stress. All pH average readings taken at the end of each cycle remain within those ranges originally detected, since the small variations found can be explained by with the normal internal variations in the paper support in relation to the surrounding environment. All samples reacted in the same way to the various extreme environmental conditions, finding a thermohygrometric equilibrium that does not alter their internal status and allows them to maintain the pH at levels optimal for a good conservation. Even the presence of inks of various kinds doesn't modified the properties of *Funori* extract, and above all the characteristics of the graphic mediations themselves, since the latter don't show any optical-chromatic variations. The resizing with the algae extract seems to be able to create an optimal internal environment both for the inks and for the paper itself, as well as to guarantee the paper artefact a good physical-mechanical stability. Considering that these results were

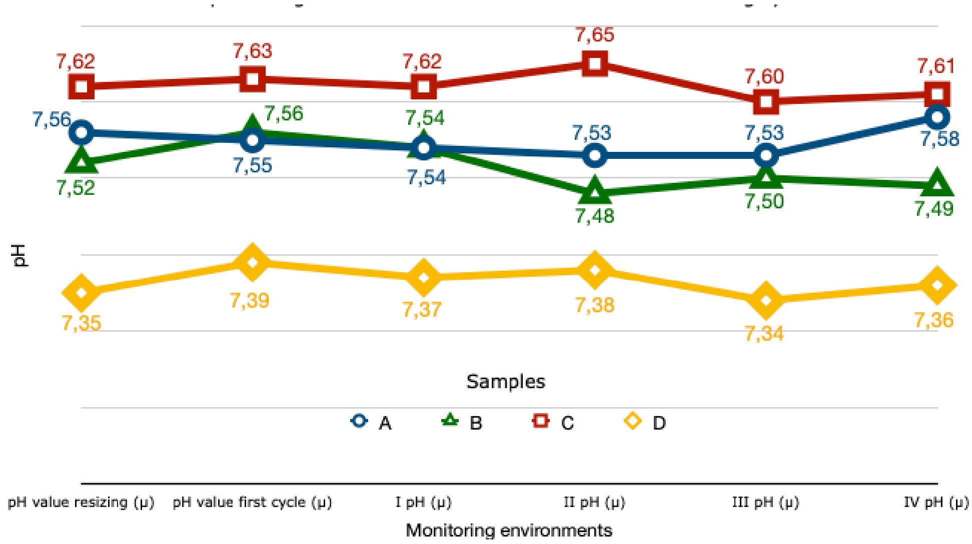


Graphic 1. pH average values trend during the first environmental monitoring cycle

In an extremely non-conservative conditions, we can deduce that *Funori* adhesive can attain similar results in environments with conservative standards. After four years of monitoring, both ancient and modern manuscript papers still show the initial tactile properties: no roughness, or stiffness appeared more or less marked, as well as no alterations to inks. At the same time, the internal environment was still settled on the original values (Table 4, Graphic 2).

				pH average values (µ) of the fourth environmental monitoring cycle			
Samples	***Funori* solution %**	**pH value after resizing (µ)**	**pH value after the first cycle (µ)**	**I 10°C + 40% RH Ph ±**	**II 25°C + 40% RH Ph ±**	**III 10°C + 60% RH Ph ±**	**IV 25°C + 80% RH Ph ±**
A	0,3	7,56	7,55 Δ[-0,01]	7,54 Δ[-0,02]	7,53 Δ[-0,03]	7,53 Δ[-0,03]	7,58 Δ[+0,02]
B	0,3	7,52	7,56 Δ[+0,04]	7,54 Δ[+0,02]	7,48 Δ[-0,04]	7,50 Δ[-0,02]	7,49 Δ[-0,03]
C	0,3	7,62	7,63 Δ[+0,01]	7,62 Δ[±0,00]	7,65 Δ[+0,03]	7,60 Δ[-0,02]	7,61 Δ[-0,01]
D	0,3	7,35	7,39 Δ[+0,04]	7,37 Δ[+0,02]	7,38 Δ[+0,03]	7,34 Δ[-0,01]	7,36 Δ[+0,01]

Table 4. pH average measurements of handwritten supports on handmade paper with iron-gall inks (A-B-C) and machine-made paper with Indian-Ink (D) after four years of environmental monitoring



Graphic 2. pH average values trend during the fourth environmental monitoring cycle

Conclusions

A *Funori* solution guarantees: high penetration inside paper fibres with reacquisition of the correct physical and mechanical properties (elasticity, flexibility, softness) of the support on which it's spread, due to its low viscosity and low surface tensions, without altering the original thickness of the object; good adhesive properties and, above all, excellent cohesive properties, capable of keeping the cellulosic fibres bounded together, due to *Funori*'s ability to absorb and bond large amount of water thanks to the ability of *funorans* to create hydrogen bonds; solubilisation and reversibility in water; weakly alkaline neutral pH; no optical properties alteration of the surface, no yellowing translucent areas or tidelines appeared; supposed antimicrobial activity. These results confirm our thesis about the suitability of the *Funori* seaweed extract for the operations of resizing paper materials.

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