ISSN 1330-3651 (Print), ISSN 1848-6339 (Online)
https://doi.org/10.17559/TV-20210831164929
Original scientific paper

The Efficiency of the Separation of Impurities from Cellulose Pulp Obtained from Pharmaceutical Laminated Cardboard Packaging

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Abstract: On the market there are increased quantities of laminated cardboard packaging with glossy surface. Laminated packaging is being used for its appealing appearance. However, laminated paper treatment creates additional problems in the process of recycling of used product. Adhesives and foil are a specific issue in the separation of impurities in recycled paper. It is crucial that the paper packaging products contained in the paper for recycling are repulpable within standard operating time and equipment. In this research three methods (without chemicals, INGEDE 11 and their combination) of removing impurities from paper pulp are compared and their efficiencies are determined to gain deeper insight into the recycling process. The procedures determine the recyclability of existing printed material, as well as the possible adhesive and material improvements. A specific issue in the separation of impurities was the presence of adhesives as well as silver foil (BOPET metallized film) for lamination. All three applied procedures successfully separate impurity particles from paper pulp and printed laminated boxes in a more sustainable manner, without the use of chemical agents.

Keywords: adhesives; deinking; flotation; *L* a* b**; paper and cardboard; pharmaceutical packaging; pulp; recyclability; stickies

1 INTRODUCTION

The evolution of the pharmaceutical industry has had an impact on people's lives because it has contributed, and still contributes to generating well-being in terms of health and quality of life for individuals, and because it is one of the most relevant sectors for the economy at a global level [1]. The pharmaceutical industry has been the subject of growing attention for the impact in terms of sustainability of its activities. Concerns about the introduction of sustainability practices into waste recycling, the reduction of water usage, greener manufacturing methods, and recyclable packaging have intensified attention on this topic. The sustainability of the pharmaceutical industry has also aroused the interest of scholars from various disciplines, such as chemistry, engineering, and environmental sciences [2].

Paper recovery rates continue to increase each year. The American Forest & Paper Association has launched its Better Practices Better Planet 2020 initiative, establishing an ambitious goal of 70% paper recovery by 2020 (the recovery rate was 63.5% in 2010) [3]. However, the use of the recycled paper is still limited by the presence of many kinds of contaminants, which can be classified according to their source, i.e. organic, inorganic and microbiological ones [4].

It is crucial that the paper packaging products contained in the paper for recycling are repulpable within standard operating time and equipment; otherwise too much material is lost and cannot be integrated in the recycled pulp. It is important that the resulting recycled pulp is optically and mechanically homogeneous, which ensures recycled pulp for high quality products. Also, it is important that adhesive impurities do not lead to microstickies at all nor to a macrosticky area that is too big. The most important parameters are therefore repulpability, yield of fibrous material, coarse reject, flake content, stickies and technical quality [5].

Paper sheets are used widely for many purposes. The production over the entire world depends mainly on forest tree pulps, which adversely affects the environment [6]. During traditional pulping processes many hazardous chemicals which damage the environment are used to produce pulp. The technological process of waste paper deinking is composed of four basic units: (1) defibering (preparation of used paper suspension), (2) washing or flotation (removal of impurities from the suspension), (3) bleaching of fibers and (4) treatment of the process water which is used in the recycling of the paper [7, 8]. Deinking is the most important step in waste paper recycling [9-11].

On the market there are increased quantities of laminated cardboard packaging with glossy surface. Laminated cardboard packaging creates additional problems in the recycling process of the used product. The number of sticky particles, but also the shiny particles is greatly increased. In our research typical papers are characterized and compared the particles of impurities on the surface of the handsheets made from recycled fibres obtained from cardboard and laminated cardboard packaging [12].

The heterogeneous nature of the produced wastes depends on the type of recycled paper and on the kinds of process units from which they were obtained. Accepted fractions should be as rich as possible in cellulose fiber up to the paper machine, thus becoming incorporated in the papermaking process [13]. Secondary fibers are the principal feedstock for paper production worldwide due to environmental and economic issues, regarding the use of recycled materials rather than pure cellulose. It was reported that 40% of total paper production is based on the use of such fibers [14].

Various methods are available for the quantification of stickies and can be classified into methods that measure the quantity, composition, or deposition tendency [15]. There are also morphological determinations using screening and microscopic analysis, which can be helpful for macrostickies. In the case of colloidal and microstickies, chemical analysis based on solvent extraction procedures and gravimetric analysis can be used. There are also reports on thermogravimetric analysis (TGA), Fourier-transform infrared spectrometry (FTIR), and nuclear magnetic resonance spectrometry (NMR) studies [16]. When the water fraction is evaluated, it is possible to use a fluorescent counting method [15], or turbidimetry.

However, there is no reliable and repeatable method that is accepted as standard.

The heterogeneous nature of organic contaminants trapped in recycled paper requires an integrated chemical approach for their complete characterization [18] and for the evaluation of their subsequent alternative uses. Some of the most commonly adopted methods of analysis are reported in the literature [19, 20]. Up to now, these methods have been mainly focused on the macrostickies analysis, but there is no generally accepted standardized method for microstickies determination.

The present paper compares three methods for paper recycling by defining recyclability of laminated paper containing adhesives, to determine the efficiency of the methods and to give an insight to the optimization of the recycling process. The research contributes to better understanding the characteristics of the obtained secondary fibers, as well as the classes of impurities which are mainly contained in the cardboard pulp obtained from laminated cardboard for packaging of pharmaceutical products. The aim of the author is to conduct research to determine which process is most effective for separating impurities from the cellulose pulp of laminated cardboard while reducing the negative effects on the environment.

2 EXPERIMENTAL

In this study, laminated cardboard printing substrate, labelled here as S, and printed pharmaceutical laminated cardboard packaging, labelled here as P, are used. Printed substrate was the standard sample for determining the efficiency of the deinking process. The printing substrate was GC2 cardboard, to which a dispersion based on acrylic polymers was applied. These self-crosslinking acrylics are free of plasticizers and alkylphenol ethoxylates (APEO). The function of the dispersion is to apply a biaxially-oriented polyethylene terephthalate (BOPET metallized) film to obtain a laminated packaging material. The BOPET metallized film complies with the repealed Directive 20/590/EEC. Printing samples were made with a standard printing form on a five-color offset machine, Roland 705. The prints were prepared with UV offset inks produced by Sun Chemical® Europe. The printing process started with white offset printing ink, continued with CMYK separation and a dark purple-blue pantone color was used at the end of the printing process for the text on the packaging. The prints were varnished with a UV-cured varnish, which is a highly reactive photopolymerizable acrylate system, VOC free, with reduced odor and optimal wetting properties. The aforementioned UV-cured varnish is VP 1038 high gloss, VergamGH (marked L2). For assembling the packaging, an adhesive was applied to the edges in compliance with the European framework directive 89/109/EEC, specific rules for adhesives in food applications and regulation (EC) of the European Parliament and of the Council on materials and articles intended to come into contact with food and repealing Directives 80/590/EEC, and Commission Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food.

After conversion of the sample into cellulose pulp (decomposition process) according to ISO 5263-2: 2004 [21], separation of impurity particles was performed in three ways.

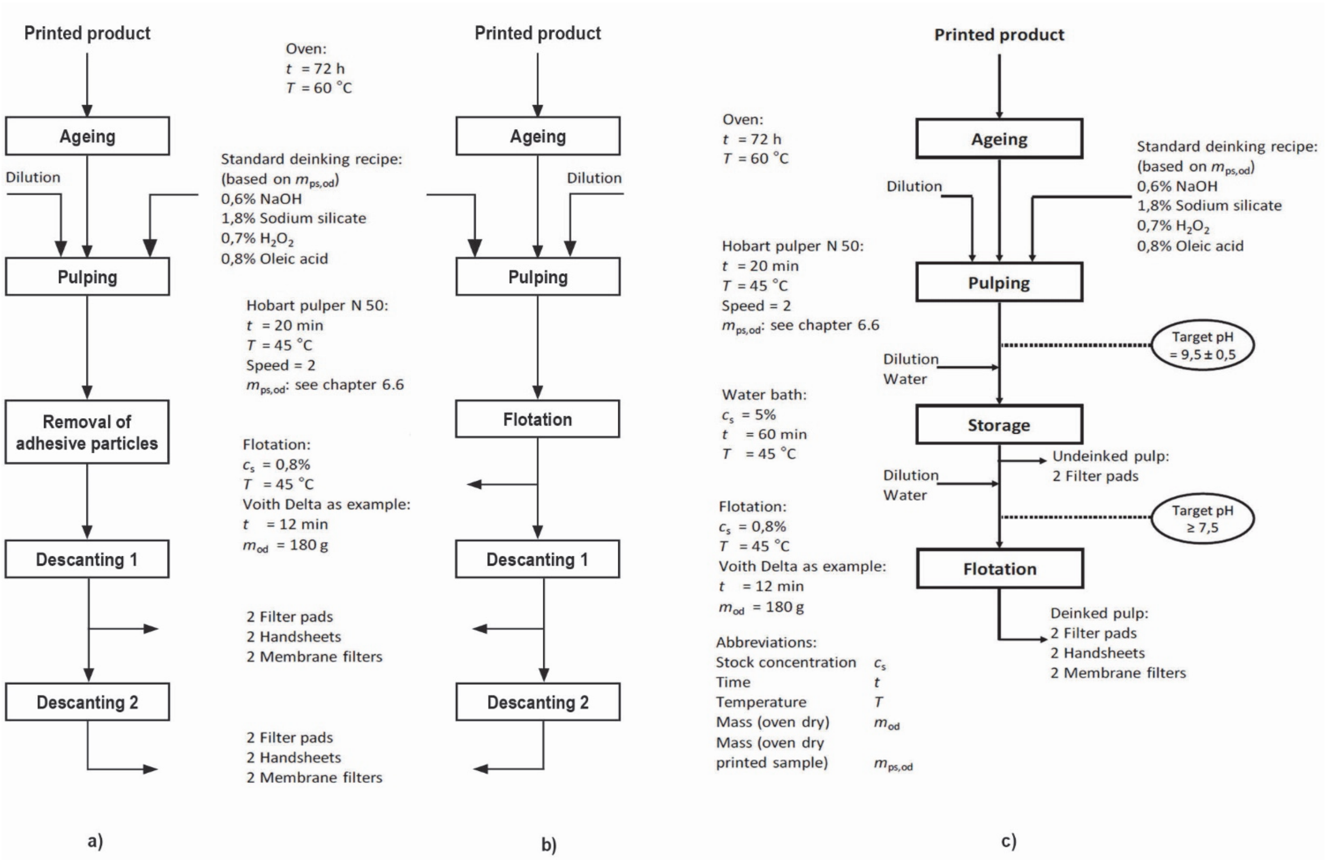

Figure 1 The flow diagram of a) a method without the utilization chemicals b) a combined method c) and the INGEDE Method 11 [22]

The standard INGEDE method 11 of deinking flotation was used as a test framework against which laboratory sheets of paper obtained by other methods were compared. The flow diagram of the process in the production laboratory sheets is shown in Fig. 1c. The mentioned process was compared with a method that does not use chemicals in the separation of impurities in the paper pulp, the P1 procedure (Fig. 1a). The intention of this process is to increase the sustainability of the deinking process, reduce the amount of chemicals used in the process (economic benefit) while obtaining a raw material that is qualitatively satisfactory. The next method, method P2, is a combination of standard INGEDE method 11 [22] and method P1 (Fig. 1b). The aim of this procedure was to see if the additional process would contribute to a higher quality of paper pulp. Higher pulp quality can contribute to the absence of a bleaching process, which ultimately results in less chemical use and reduced impact on the environment. Manual sheets are made according to standard INGEDE 1 procedure [23] and ISO 5269-2: 2004 [24] standards, in all phases from P1, P2 and P3. Procedures using Rapid Köthen sheet former.

The following methods were used for measuring the optical characteristics of laboratory handsheets: the diffuse blue reflectance factor according to ISO 2470-1:2016 [25], effective residual ink concentration, ERIC according to TAPPI T 567:2009 [26], ISO 22754:2008 [27] and color determination for paper and board, ISO 5631-3:2015 [28]. Image analysis was used to count the detected residual impurity particles and area. Spec × Scan Apogee System image analysis software according to ISO 13322-1, 2014 [29] was used, which includes a scanner to digitalize images. The values on the device were as follows: threshold value (100), white level (75) and black level (65) were chosen after comparing the computer images to the handsheets.

3 RESULTS AND DISCUSSION

By studying the results of the ISO brightness measurement of the handsheet samples obtained from printed boxes and printed media after the separation of impurity particles, it is observed that all the values increase following this procedure. Therefore, it can be concluded that all the procedures contributed to an increase in brightness and the impurity particles had been separated. The highest measured values were obtained using the INGEDE Method 11, which is the standard method for recycling paper and cardboard.

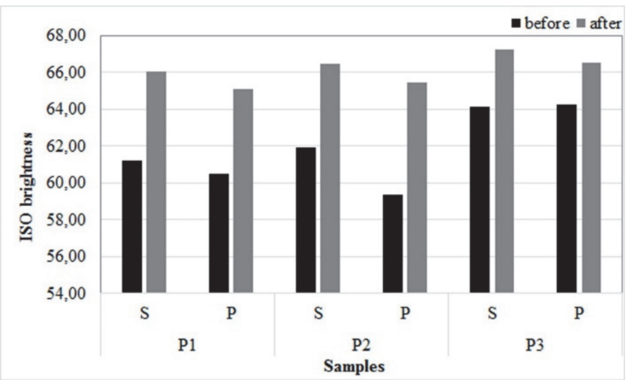

Figure 2 The effect of the deinking process on ISO brightness

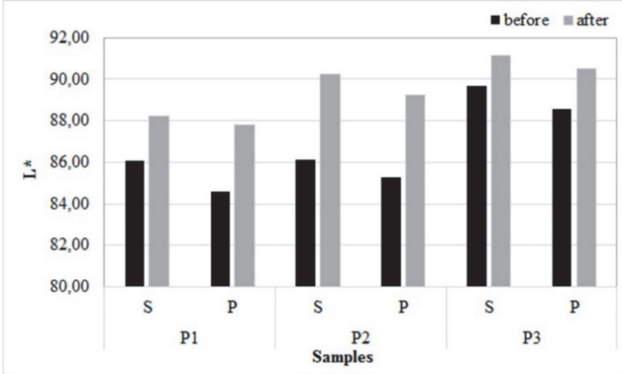

Figure 3 The effect of different impurity separation procedures on the values of the colorimetric coefficient *L**

Unlike earlier interpreted results, the values of the colorimetric coefficient *L** show the influence of the reflection of color as a combination of tint, saturation and dark/light value. *L** measures are the lightness of the samples. The trends of the measured values of the handsheets before and after the impurity separation process follow the trends measured for ISO brightness and contribute to the confirmation of the previously measured results.

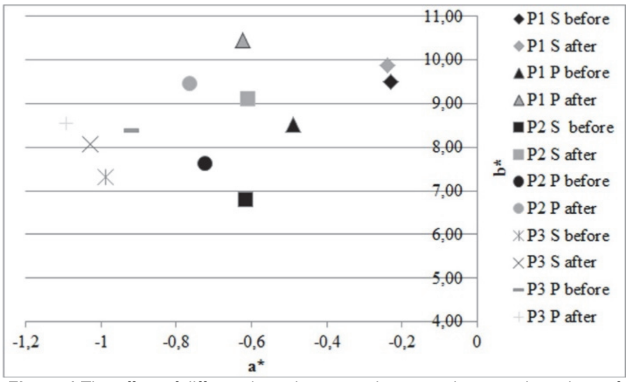

Figure 4 The effect of different impurity separation procedures on the values of the colorimetric coefficients *a** and *b**

When the values of the colorimetric coefficient *b** of the handsheets obtained from printed laminated substrates are examined, it is evident that the optical brighteners are separated after the separation of impurities because the handsheets colorimetrically go toward yellow tones. It should be explained that in the first and second decantation processes, in addition to dye particles, optical brighteners are also rinsed off.

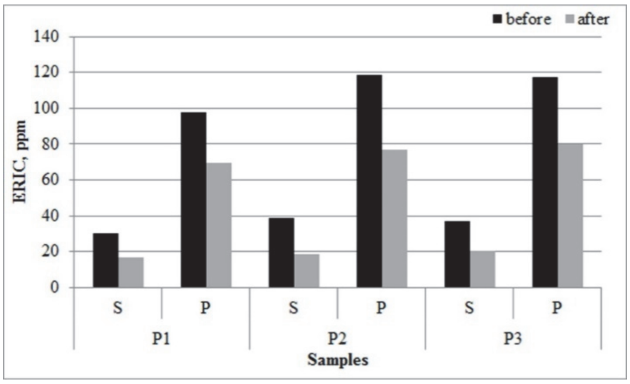

Figure 5 The effect of different procedures for separating impurity particles on the ERIC value

The same trend is also present for samples obtained from printed laminated boxes, although the value coefficient *a** decreases, which means that the sheets acquire a slightly bluish tone. The reason for this phenomenon is that the printing on the box is blue-purple. This trend is most noticeable with the standard INGEDE Method but it is important to mention that these samples lose the least optical brighteners and do not have a greatly increased *b** value.

By measuring samples of handsheets obtained from printed substrates with Procedure P2 prior to the separation of impurity particles, the highest values of particles were obtained but it can be seen that the measured values after the separation of the impurity particles were the lowest. Thus, it could be concluded that in Procedure P2, the largest quantity of particles generated are those that are best separated by the processes in the procedure. In general, it can be concluded that the quantity of particles on the handsheets obtained from printed substrates is small and they are efficiently separated by all the procedures. Good efficiency in the separation of impurity particles is also seen for printed laminated drug boxes. From the measured ERIC values, which are at most 80 ppm, it can be concluded that the obtained hand sheets are of satisfactory optical quality for the production of packaging products.

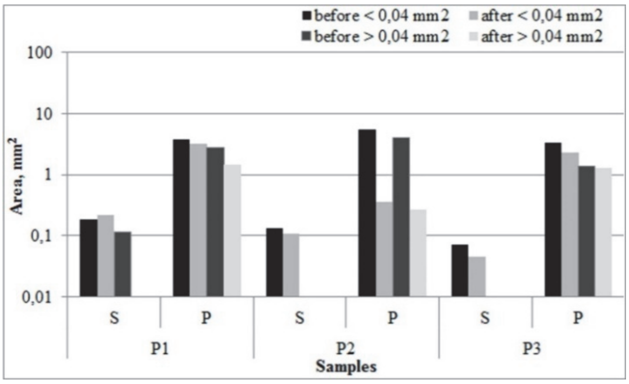

Figure 6 The effect of different procedures for the separation of impurity particles on the particle surfaces on handsheets

By studying the surfaces of the impurity particles left on the handsheets before and after the particle separation procedure for the samples of laminated print substrates, it can be established that the separation process is highly successful since, after the procedure, generally no impurity particles are presented. In Procedure 1, after the process of separating the impurity particles obtained from the substrate, the particle surfaces in the class of particles < 0,04 mm^2 increased. The reason for this phenomenon is that some of the particles become fragmented into smaller particles from the class of particles > 0,04 mm^2. It must be noted that the total surface of all the impurity particles after the particle separation process decreased by 40%. Comparing the procedures for the samples of printed laminated boxes, Procedure 2 reduces the surface area of the impurity particles of the handsheets the most after the separation of the impurity particles in both particle size classes. This method is particularly distinguished for the separation of particles belonging to the class > 0,04 mm^2. Such particles affect the inhomogeneity of the sheets of paper and are usually more difficult to separate using the conventional procedure, which can be seen in comparison to Procedure 3, where there is the least separation of such particles.

The number of particles on the handsheets after the procedure for the separation of impurity particles in both classes is generally zero. The only exception occurs in Procedure 1 in the particle size class of < 0,04 mm^2, where two particles are left. In this case, the number of particles was reduced fifteen-fold, so that even then the procedure is efficient. Given the small number of particles, it can be concluded that the particles in this class do not significantly affect the grayness of the paper. When studying handsheets made from the pulp of printed boxes before and after the separation of impurity particles, it is seen that Procedure 2 is the most efficient, where the number of particles in both classes is reduced approximately ten-fold. The remaining two procedures are equally successful in separating particles and the number of impurity particles is reduced by about half of the initial values.

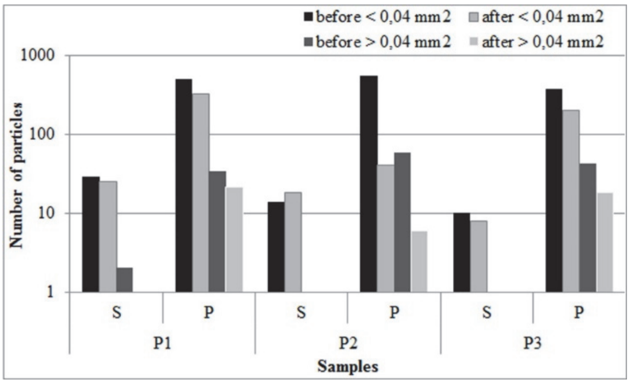

Figure 7 The effect of different impurity particle separation procedures on the number of particles on the handsheets

4 RESULTS AND DISCUSSION

Regarding the fidelity of the ISO brightness and colorimetric coefficient *L**, the greatest increase in value is seen with Procedure 2, which can be explained by the removal of the largest number of impurity particles. The same conclusion can be confirmed from the lowest ERIC values. It is nevertheless important to note that with Procedure 3, the highest ISO brightness and colorimetric coefficient CIE *L** is achieved, from which it can be concluded that this method removes the fewest optical brighteners from the samples. This fact is also confirmed by the values of the colorimetric coefficient CIE *b**, which are the lowest but higher than zero, i.e., they are in the brightest yellow range in comparison to the other samples.

By studying the number of particles on the handsheets before and after the separation of the impurity particles, it can be noted that Procedure 2 is the most successful, which is a combination of two methods for separating impurity particles. The separation performance of Procedures 1 and 3 are equal but it is significant that chemicals are not used in Procedure 1, which increases the sustainability of this method. The most successful procedure for separating impurity particles > 0.04 mm^2 is Procedure 1. This can help explain the efficiency of Procedure 2. The good fragmentation of the class of particles > 0,04 mm^2 in Procedure 1 and the presence of INGEDE deinking chemicals in Procedure 3 easily separate the formed

impurity particles from paper pulp and yield the best separation results. It is important to note that by examining the optical homogeneity of the handsheets achieved by all the procedures, there are no inhomogeneities on the handsheets, according to S. Runte et al., who state that such samples have very good visual quality [30].

From this study, it can be concluded that all three methods satisfactorily remove impurity particles from pulp made from printed laminated substrates and printed laminated boxes for drugs. A specific issue in the separation of impurities from the said samples is the presence of adhesives as well as silver foil (BOPET metallized film) for lamination. There is a higher concentration of adhesive in the pulp when the adhesive is applied to the entire surface of the cardboard or the printed substrate but the adhesive used for gluing boxes should also be mentioned. All of the above contribute to the complexity of the process of separating impurity particles from paper pulp because different types of particles are formed (including stickies), many of which can agglomerate due to the presence of adhesive. The present study contributes a procedure that successfully separates impurity particles from paper pulp in a more sustainable manner, without the use of chemical agents.

5 CONCLUSION

Regarding the fidelity of the ISO brightness and colorimetric coefficient L^*, the greatest increase in value is seen with Procedure 2, which can be explained by the removal of the largest number of impurity particles. The same conclusion can be confirmed from the lowest ERIC values. It is nevertheless important to note that with Procedure 3, the highest ISO brightness and colorimetric coefficient CIE L^* is achieved, from which it can be concluded that this method removes the fewest optical brighteners from the samples. This fact is also confirmed by the values of the colorimetric coefficient CIE b^*, which are the lowest but higher than zero, i.e., they are in the brightest yellow range in comparison to the other samples. By studying the number of particles on the handsheets before and after the separation of the impurity particles, it can be noted that Procedure 2 is the most successful, which is a combination of two methods for separating impurity particles. The separation performance of Procedures 1 and 3 is equal but it is significant that chemicals are not used in Procedure 1, which increases the sustainability of this method. The most successful procedure for separating impurity particles > 0.04 mm^2 is Procedure 1. This can help explain the efficiency of Procedure 2. The good fragmentation of the class of particles > 0,04 mm^2 in Procedure 1 and the presence of INGEDE deinking chemicals in Procedure 3 easily separate the formed impurity particles from paper pulp and yield the best separation results. It is important to note that by examining the optical homogeneity of the handsheets achieved by all the procedures, there are no inhomogeneities on the handsheets, according to S. Runte et al., who state that such samples have very good visual quality [30].

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