



ANTIVIRAL COATINGS TO PRODUCE BREATHABLE AND BIODEGRADABLE FILTER MASKS

REVESTIMENTOS ANTIVIRAIS PARA PRODUZIR MÁSCARAS COM FILTROS RESPIRÁVEIS E BIODEGRADÁVEIS

RECUBRIMIENTOS ANTIVIRALES PARA PRODUCIR MASCARILLAS FILTRANTES TRANSPIRABLES Y BIODEGRADABLES



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ABSTRACT

Surgical masks are recommended by the World Health Organization for personal protection against disease transmission. However, most of the surgical masks on the market are disposable and cannot be self-sterilized for reuse. Thus, when confronting the global public health crisis, severe pollution due to disposable masks is inevitable. In this paper, a novel low-cost filter mask with excellent breathability is reported to replace the filters' oil derivatives through an ecologically correct filter with properties sustainably optimized on a pre-industrial scale (roll-to-roll). A roll of common paper was passed through a vacuum chamber and deposited by CVD. Hydrophobicity and permeation were measured through contact angle, SEM and AFM. The droplets do not penetrate the filter for a minimum of two hours. Moreover, the filters proved to be efficient in antimicrobial and breathability tests.

Keywords: Antiviral Coating. Filter Mask. CVD.

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RESUMO

Máscaras cirúrgicas são recomendadas pela Organização Mundial da Saúde para proteção individual contra a transmissão de doenças. No entanto, a maioria das máscaras cirúrgicas no mercado é descartável e não pode ser autoesterilizada para reutilização. Assim, ao enfrentar a crise global de saúde pública, a poluição severa devido a máscaras descartáveis é inevitável. Neste artigo, uma nova máscara de filtro de baixo custo com excelente respirabilidade é relatada para substituir os derivados de óleo dos filtros por um filtro ecologicamente correto com propriedades otimizadas de forma sustentável em escala pré-industrial (rolo a rolo). Um rolo de papel comum foi passado por uma câmara de vácuo e depositado por CVD. A hidrofobicidade e a permeação foram medidas por ângulo de contato, SEM e AFM. As gotículas não penetram no filtro por um mínimo de duas horas. Além disso, os filtros provaram ser eficientes em testes antimicrobianos e de respirabilidade.

Palavras-chave: Revestimento Antiviral. Máscara com Filtro. CVD.

RESUMEN

Las mascarillas quirúrgicas son recomendadas por la Organización Mundial de la Salud para la protección personal contra la transmisión de enfermedades. Sin embargo, la mayoría de las mascarillas quirúrgicas en el mercado son desechables y no pueden autoesterilizarse para su reutilización. Por lo tanto, al enfrentar la crisis mundial de salud pública, la contaminación severa causada por las mascarillas desechables es inevitable. En este artículo, se informa sobre una novedosa mascarilla con filtro de bajo costo con excelente transpirabilidad que reemplaza los derivados del petróleo de los filtros a través de un filtro ecológicamente correcto con propiedades optimizadas de forma sostenible a escala preindustrial (rollo a rollo). Un rollo de papel común se pasó a través de una cámara de vacío y se depositó por CVD. La hidrofobicidad y la permeación se midieron mediante ángulo de contacto, SEM y AFM. Las gotas no penetran el filtro durante un mínimo de dos horas. Además, los filtros demostraron ser eficientes en pruebas antimicrobianas y de transpirabilidad.

Palabras clave: Recubrimiento Antiviral. Mascarilla con Filtro. CVD.



1 INTRODUCTION

The coronavirus pandemic of 2019, abbreviated as COVID-19 [1,2], has been considered an unprecedented healthcare crisis since the Spanish Flu pandemic in the early 20th century, which severely disrupted nearly every aspect of daily life. As of March 2023, the World Health Organization (WHO) has reported over 760 million confirmed cases of COVID-19, with a death toll of over 6.8 million. The primary cause of this disease is the severe acute respiratory syndrome coronavirus (SARS-CoV-2), which causes infected individuals to manifest flu-like symptoms [3,4]. The general transmission pathway for COVID-19, like other respiratory diseases, consists of contact transmission and aerosol transmission. Face masks or respirators can be an effective and essential equipment to protect healthcare workers and members of the general public who may be exposed to the virus. [5]

Disposable surgical masks can prevent respiratory droplets from entering the lungs, which can help to reduce the risk of getting infected, alongside proper hand hygiene and cleaning methods. [6-11] However, there are some limitations to existing masks. These include the difficulty of recycling and reusing masks, as the captured viruses might stay on their surfaces.

In 2020, the daily worldwide production rate of surgical masks was over 40 million pieces, resulting in more than 15,000 tons of waste every day. Even today, thousands of tons of masks are discarded. In order to process these used masks, most countries can only use incineration to treat such medical waste. The resulting toxic gases and high carbon emission greatly harm the environment.[12]

It is extremely important to manufacture masks and filters for reusable masks that have the appropriate characteristics to block the severe acute respiratory syndrome coronavirus, that are biodegradable and disposed of in an ecologically correct way. Simple replacing the PP-based mask (nonwoven masks) by various types of cloth masks with filters can address the issue related to the biodegradability pollution.[13] In this sense, solutions are needed to supply the entire market with a biodegradable and antiviral product. For several reasons, cellulose is one of the most promising alternative materials that can be used to replace synthetic fibers traditionally used as filter and disposable masks. These reasons include its biodegradability, very low cost, renewability, abundance, strong mechanical properties, low density, and environmental friendliness compared to other materials. [14]

In general, face masks and respirators consist of three layers: a middle filter layer – the most important layer with regards to protection from particles or droplets carrying viruses and bacteria, and two other layers. It has been reported that using a multi-layer mask has improved filtration compared to one-layer tissue masks. However, filtration efficiencies of the

hybrid fabrics were larger than 80% for particles <300 . Macroparticles (diameters $> 0.6 \mu\text{m}$, e.g. polluted aerosols and most bacteria) are generally larger than pore sizes of mask filters and can be transported outside of face masks. Microparticles ($\sim 0.3\text{--}0.6 \mu\text{m}$, e.g. some large bacteria and viruses) have a chance to move through pores, but hardly reach users. However, nanoparticles ($<0.3 \mu\text{m}$, like viruses) can easily flow through pores and do not adhere to fiber walls. [15] For this reason, reusable mask filters that completely inhibit the passage of microorganisms become important. Hydrophobic and antimicrobial filter based on cellulose becomes an effective solution to this demand.

So far, many methods have been adopted to produce hydrophobic coatings on cellulose-based materials, including various dip-coating methods, in situ nanorod/particle growth, spray coating, chemical vapor deposition (CVD), and plasma processing techniques. [16]

For reducing the spread of the virus, hydrophobic and antimicrobial surfaces play a significant role. In view of this, the present article focuses on manufacturing effective filters, made from common paper, with surface treatment, in controlling the COVID-19 virus and its antiviral property. The wettability of the treated papers was characterized by water contact angle (WCA) and the surface morphology was observed by scanning electron microscopy (SEM) and Atomic Force Microscopy (AFM). Besides, the breathability and antimicrobial property have been tested.

2 EXPERIMENTAL DETAILS

2.1 COATINGS PREPARATION

Specimens of common paper were used as the substrate in this study. The coatings were prepared in a semi-industrial production process. The roll-to-roll high-vacuum device is equipped with two titanium chambers measuring (100×150) mm. The substrate is guided between the chambers, and a nanolayer is deposited by CVD [17].

Four different samples were prepared. Samples are identified as P (Paper), P + Ti (Titanium), P + Ti + CO₂ and P + Ti + CO₂ + SO (HMDSO). A roll of common paper (toilet paper) is fitted into one of the two chambers of the reactor and stretched to the second chamber's roll. The chamber is closed, and a mechanical pump is turned on for vacuum (base pressure 3×10^{-3} mBar). Samples P + Ti were prepared by introducing 10 sccm of argon in the chamber. For P + Ti + CO₂ samples, 10 sccm of CO₂ (carbon dioxide) is inserted until the pressure reaches a value of 6×10^{-2} mBar. When P + Ti + CO₂ + SO (HMDSO) samples were made the container with HMDSO is opened until a pressure of 1.5×10^{-1} mBar is reached. An external DC source with a voltage of 20 W powers two titanium rectangular

targets so that an electric arc opens to vaporize material from a cathode target, the plasma opens, and the deposition of precursors takes place. The total deposition process takes 20 minutes. After deposition, the coiling system is turned on (80 cm/min) for new deposition to occur. For comparison, silicon oil, titanium, and carbon dioxide were used as coating precursors. With the roll-to-roll system presented in Figure 1.

Figure 1

Roll-to-roll system with toilet paper roll



Can be possible to deposit meters of filters at once, which makes the process faster, in addition to being a simple and economical process. Once the vacuum is established, successive depositions can be made on the entire inserted roll.

2.2 MATERIAL CHARACTERIZATION

2.2.1 Surface morphology

The surface morphology of paper coated was analysed by scanning electron microscope (SEM) and atomic force microscope (AFM). For the SEM, the Bruker Nano was used with a constant acceleration voltage of 10 kV, a working distance of 10 mm, and room temperature. A piece of paper sample coated with silicone oil was cut, attached to a sample holder with carbon tape, and covered with silver (sputtering) for 250 seconds at 30mA. AFM analyses were performed on the JPK Instruments model 1 M plus microscope. The high-resolution images of the surface were obtained in air, at room temperature, in intermittent contact mode, setpoint 0.5V and speed 1Hz. Bruker needles with a spring constant of 1-5 N/m and a frequency of 66.85 KHz were used.



2.2.2 Contact angle analysis

The hydrophilicity of the samples was measured through the contact angle using the Ramé-Hart NRL A-100-00 goniometer, operated in air and room temperature. To carry out this analysis, a drop of Mili-Q water was dripped onto three distinct regions of the film and the evolution of the droplet's behavior on the surface was measured at an interval of 0.2 seconds for a total time of 500 seconds.

2.2.3 Breathability test

To select materials with resistance compatible with N95 masks available on the markets and with breathability, the wPWG System was used, consisting of an air syringe precisely moved by a servomotor, controlled by the Pulmonary Waveform Generator System software. The wPWG system allows fine flow control and shaping of the inspiratory (Insp) and expiratory (Exp) waveform, pause times, number of cycles, and accurate measurement of pressure and air flow within the system. It has a capacity of 0-12 Liters of volume, a flow rate of 0-16 L/sec, with acceleration of 0-1200 L/sec².

By placing different types of materials at the syringe outlet, an increase in resistance is generated, which can be estimated by the increasing pressure in the system during the Insp/Exp cycles. Comparing the pressure values obtained using different fabrics and papers with those obtained from N95 masks available on the market, we can select the most suitable materials for making masks based on their air permeability.

For the tests, a square wave of mirrored flow was used for Inspiration and Expiration, with a constant flow of 0.86L/s and a rise time of 0.01s, without pauses between Inspiration/Expiration. Three cycles were generated for each material with constant pressure and flow measurements and a tidal volume of 2L. To avoid pressure variations depending on the beginning and end of the cycles, the pressure values used for the comparative analyses were measured during the first 100 milliseconds after the first second of each cycle.

Due to the elasticity of the materials, there is an intrinsic difference between the pressures measured during Inspiration and Expiration. In Inspiration the pressures are consistently higher, as the material is sucked into the syringe making a semicircular “negative” concavity that increases the resistance to the passage of air.

During exhalation, the opposite occurs, the material is expelled from the syringe, again making a semicircular concavity, now “positive”, which decreases resistance as it increases the air passage area when compared to inspiration.

2.2.4 Antimicrobial analysis

For the antimicrobial analysis, the paper without the coating and with the coating were added to sterile tubes, and 2ml of Brain Heart Infusion Broth (BHI -HIMEDICA© M210-500G) was inserted into them, which is a culture medium used to stimulate the proliferation of microorganisms. This liquid material has an originally yellowish and clear color. The turbidity that may occur in this liquid during periodic analyzes will act as a visual indicator of microbial growth. All this analysis was done at room temperature and carried out in a clean and sterile area (laminar flow Pachane, Pa410 – ECO). The test were carried out for 11 days.

3 RESULTS AND DISCUSSION

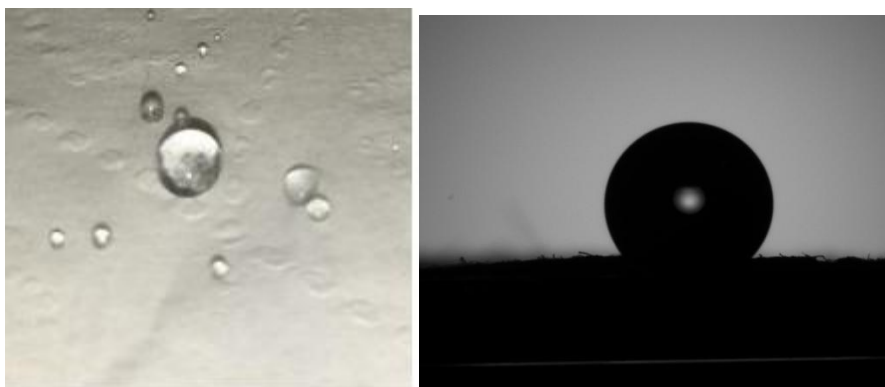
The masks are primarily used for facial protection, mainly to reduce the chance of infection through respiratory droplets generated by talking, sneezing, and coughing. They reduce the risk of breathing in these respiratory droplets and consequently avoid carrying viruses into the lungs [18].

Analyzing the surface of the treated paper, an increase in hydrophobicity was observed, with angles above $120^{\circ} \pm 0.2^{\circ}$, as shown in table 1. It was not possible to obtain the contact angle of the sample without the coating because it is hydrophilic and absorbs all the water on its surface. The samples are identified as P (Paper), P + Ti (Titanium), P + Ti + CO₂ and P + Ti + CO₂ + SO (HMDSO). The Milli-Q water drops, when dripped onto the treated samples, do not interact on their surfaces, as shown in figure 2.

Figure 2

Sample to paper after plasma treatment





The table below shows the results about contact angle after plasma treatment.

Table 1

Contact Angle Table

Sample	Contact Angle (θ) ± 0.2
P	-
P + SO	133.3
P + Ti	128.4
P + CO ₂	130.6

The contact angle as a function of time was also analyzed, and it was verified that the water drop does not penetrate the filter for a minimum period of two hours. After this time, the water droplet begins to decrease in radius, indicating that the water droplet may be evaporating, since the contact angle with the surface remains at a high value, still indicating hydrophobicity.

Filters made with treated paper showed to be hydrophobic, which means that the coating can block most of the respiratory droplets from the outside. It is important that the filter is hydrophobic and breathable, and for this, the samples were tested for breathability using the filter (treated paper) with a double-layer of tissue. The selected tissue for the production of the masks was 400 thread count cotton and half mesh for the inner and outer layers, respectively. This double-layer configuration with the filter paper (treated and untreated) between the layers was tested, and there was no significant change in pressure, which indicates that the paper doesn't alter or impair breathing.

To also understand how the coating is deposited on the surfaces, which leaves hydrophobic properties but does not alter breathability, microscopy analysis was carried out.

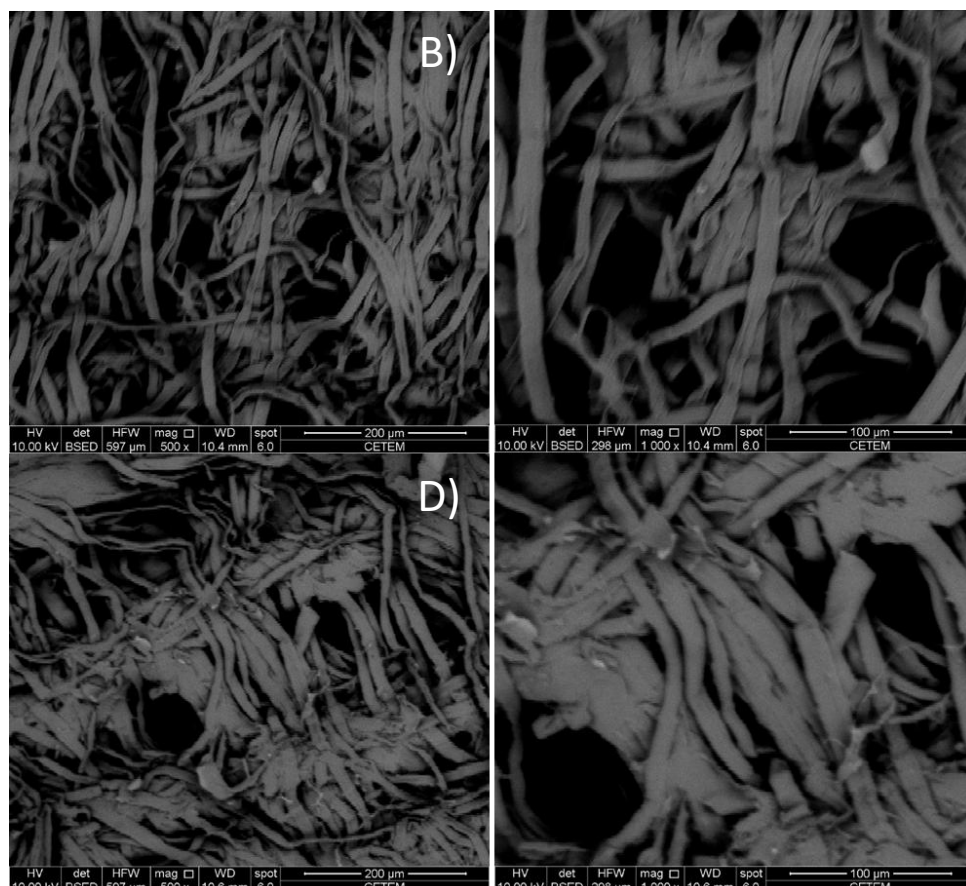
3.1 MICROGRAPHY ANALYSIS

Even though the treatment provides a coating that prevents water from passing through the sheet of paper, it does not cover its entire surface. Therefore, the breathability is not modified. The coating is present around the fibers and not as an entire film on the surface. The SEM micrograph (figure 3) illustrates the paper fibers without and with treatment (P + SO). With the help of the SEM software, it shows that the treated paper fibers have a greater thickness than the untreated paper fibers. This is due to the CVD treatment providing a hydrophobic coating to the paper surface.

The average thickness of an untreated paper fiber is 13 μm (diameter), whereas the thickness of a treated fiber is 17 μm , showing that the coating increases the fiber diameter by approximately 2 μm .

Figure 3

SEM micrograph of P + SO sample A) untreated 500x; B) untreated 1000x; C) Treated 500x and D) Treated 1000x paper fibers

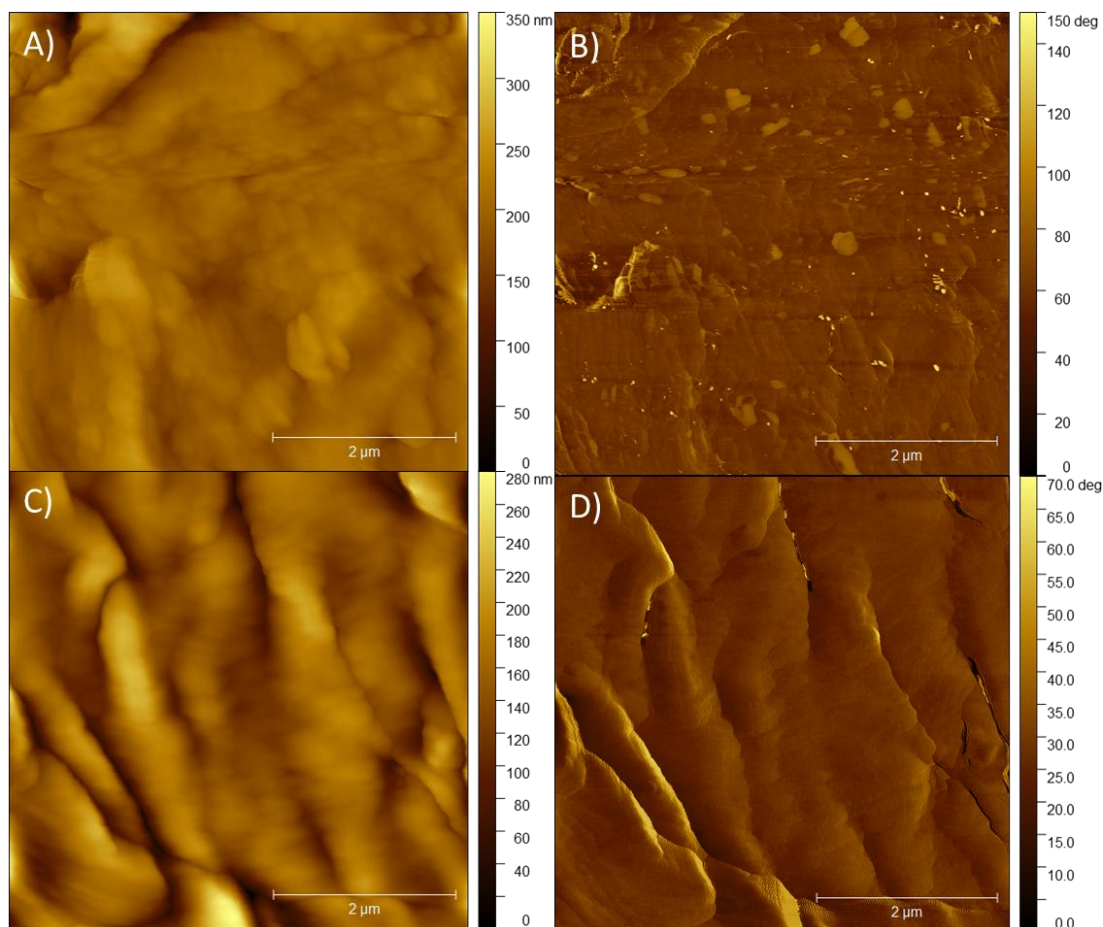


The AFM images (figure 4) show the topography and surface phase of a paper fiber, with a magnitude of $(5 \times 5) \mu\text{m}^2$, before and after treatment (P + SO).

In the image of the untreated fiber, cellulose crystals are observed, while in the images of the treated fiber, they are not so apparent. These images prove the modification of the treated surface. It is also interesting to note that the deposition was uniform, and there was no concentration of materials in specific regions.

Figure 4

AFM micrography of A) untreated height ; B) untreated phase; c) Treated height and D) Treated phase paper fibers



3.2 ANTIMICROBIAL ANALYSIS

Several clean surfaces can become infected after contact with a contaminated area and can be passed on by people who have come into contact with that surface. Furthermore, poor cleaning can leave infected particles, contributing to the spread of the disease. The use of antimicrobial surfaces can help prevent additional contamination, minimizing the occurrence of infections transmitted by touching contaminated surfaces. An antimicrobial action greatly reduced the infectivity of virus.[19]

The samples were then identified, (P; P + Ti; P + SO; P + CO₂; NO (non-woven masks made of polypropylene)). In addition to the flasks that were sealed with the samples for comparison, two more flasks were used to carry out a comparison: One considered as a negative control (NC), with the same amount of BHI and sealed with its respective lid. The second, the positive control (PC), is also a bottle with the same amount of BHI, however, it will be exposed to the external environment, that is, open.

With the help of Table 2, we can see that the paper samples coated with Ti and Ti + CO₂ are efficient in terms of long-term microbial permeability. The paper coated with titanium and carbon is ideal for preventing the permeation of bacteria and viruses. Antimicrobial activity has already been evaluated in other studies coatings based on Titanium, for example, Arik et al (2011).[20]

Table 2

Microbial test of analysed samples

Sample	Time			
	2 days	4 days	9 days	11 days
TNT	✓	-	-	-
P + SO	✗	✗	✗	✗
P + Ti	✗	✗	✗	✗
P + CO ₂	✗	✗	✗	✗

Subtitle:



There was growth of microorganisms



There was no growth of microorganisms

4 CONCLUSION

Cellulose-based filters, alternatives to disposable TNT masks, were produced, tested, and satisfactorily validated and can be applied to combat the proliferation of respiratory diseases, mainly (SARS-CoV-2).

Contact angle tests, along with SEM micrograph images, showed that the coating makes the paper hydrophobic but still breathable. It was also possible to observe that the thickness of paper fibers, when coated, become thicker. The treated papers showed better antimicrobial behaviour than the untreated papers. The titanium and carbon paper surface indicated superior antimicrobial property.

From this work, biodegradable, breathable, hydrophobic, antimicrobial and antiviral filter were manufactured in a simple, semi-industrial way and can be disposed of in an environmentally friendly manner.

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