

Introducing all-polyamide composite coated fabrics: A method to produce fully recyclable single-polymer composite coated fabrics

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ABSTRACT: Novel all-polyamide composite (APC) has been developed to replace traditional coated fabrics with good interfacial adhesion and enhanced recyclability. The composite is fully recyclable since it contains no other materials except polyamide. APC was prepared by partially dissolving a polyamide fabric by treatment with a film-forming polyamide solution. The effect of the polyamide solution concentration and gelling time on tensile and viscoelastic properties of APCs was investigated to explore the optimum processing parameters for balancing the good interfacial adhesion. The composite properties were studied by dynamic mechanical thermal analysis (DMTA), tensile testing and scanning electron microscopy (SEM). The results showed a good adhesion between the coating and the fabric. A new method was introduced to convert a low value added textile waste to a high value-added product. The composite is tunable, in terms of having a dense or a porous top-layer depending on the end-use requirements. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 42829.

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INTRODUCTION

Heterogeneous composites pose a recycling challenge¹ because of their composition. Furthermore, they often suffer from poor matrix-fiber adhesion due to the chemical incompatibility of the components.²⁻⁴ The growing interest in the recycling of materials is brought about by the desire to preserve the environment, as there is limited landfill space due to the large amount of wastes that is being dumped.⁵ Global warming is also of concern due to emissions from waste incineration.⁴ These challenges have stimulated an interest in the development of environmentally friendly materials.^{6,7} One promising approach to deal with the composites recycling challenge is the introduction of single-polymer composite (SPC).⁶ In SPCs both the reinforcing and the continuous phase are polymers with the same chemical composition⁵; therefore, recyclability is enhanced^{8,9} as no material separation is necessary. These materials are often described as one-polymer composites, homocomposites, all-(the same)polymer composites, self-reinforced, or homogeneous composites.^{4,5} Besides recyclability, the interest in the concept of SPCs is based upon the premise that interfacial bonding should improve if matrix and reinforcement are made from the same polymer.^{2,5}

Polymer-coated textiles are flexible composite materials comprising a coating (the polymer) and a substrate (textile/fabric layer(s)) adhered together through a specific coating process.¹⁰ The coating may be on one side or both sides either with the same or a different polymeric coating per side.¹¹ Coated woven fabrics are used in a broad range of semi-structural applications to provide lightweight, architecturally striking solutions.¹² They are often used for wide-span surfaces, membrane-cable structures, hanging roofs (e.g., roofs of sports structures like stadipneumatic constructions,¹³ transportation, ums), and commercial constructions.14 The combination of the different properties of each layer determines the overall properties of the system.¹⁰ Coated fabrics can be tailored to be used as a textile reactor, temporary houses and tents, facade coverings, container linings, and tarpaulins.¹⁵ Fabric structures resist environmental loads, as the tensile stresses are dispersed in the plane of the fabric.¹² This means that the textile substrate contributes to the mechanical strength (tear, tensile), elongational, and dimensional properties of the system in general; whereas, the polymer coating helps to introduce penetration resistance and impermeability (to liquids, gases, and dust particles), as well as improve fabric abrasion strength.¹⁰ They can also help to modify the appearance for decorative purposes.¹⁶ Coated fabrics are popular mainly due to their affordable prices, high strength, durability, resistance to wear and tear, good toughness, various colors, and soft texture.6

A wide range of textile materials are used as substrates for coated fabrics such as cotton, rayon, polyamide (PA), and

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polyester, depending on the end-use requirements.¹¹ A military tent can be made of a material that combines a PA fabric with a neoprene coating. For applications where high strength is required, PA and polyester are used, as they have considerably higher strength-to-weight ratios.¹¹ PA or Nylon is a well-known high-performance engineering thermoplastic with excellent properties, including high tensile and impact strength, tear and fatigue resistance, thermal stability, and low coefficient of friction.¹⁷⁻¹⁹ Because of their mechanical properties and their barrier-like behavior toward gasses and flavors, they are widely used in film products for flexible food packaging.²⁰ Because of its excellent wearing durability,¹⁷ the most important field of application for PAs is in the textile industry.²¹ In the family of PA fibers, PA66 possesses relatively better mechanical properties and is applied in the production of tires, airbags, bullet-proof vests, and so on.17

Adhesion is the state in which two surfaces are held together at an interface by forces or interlocking action or both.²² Adhesion between different components in composites is an important parameter, to achieve good composite properties. Poor adhesion results in failure caused by the insufficient stress transfer between the matrix and the reinforcing fabric (as the reinforcement). This is also the case when producing coated fabrics, to get a good adhesion between the coating and the fabric; moreover, it is necessary to select the right coating binder that can bind the coating to the fabric. There are several ways to increase the adhesion (or hold the surfaces together at interface by forces or interlocking action or both); however, they are often associated with drawbacks like high cost, formulation complexicity, or fabric degradation (in order to be bonded to the binder), etc.

One possible method to increase the adhesion in PA-coated fabrics could be using the same material in the coating of the PA fabric and preparing a SPC-coated fabric. By using this method, the surface of the fabric will be partially dissolved in order to entangle the polymer chains of the coating (as a continuous phase of the composite) with the dissolved polymer chains of the fabric's surface. Since the two components (fibers of the fabric and the coating) are the same, and the same solvent (formic acid) exists between them, the adhesion (which in this case is a non-reactive adhesive) would be very strong. In addition, there is no need to use any other material except PA and the solvent. By this method, novel All-Polyamide Composites (APC) coated fabric, a special form of SPC, can be obtained. APCs are composites in which both constituents are made from one kind of polymer. Furthermore, they are easily recycled, as they are composed of one and the same polymer. A number of techniques have been used or developed to prepare SPCs, including film stacking, powder or solution impregnation, hot compaction, coextrusion, and selective surface dissolution.²³ The reports about APC in the literature describe the use of two different PA grades with different melting points that are used to produce a laminate,^{17,24,25} or show the preparation of all-aramid composites by partial fiber dissolution.²⁶ However, there is no report about producing SPC-based PA-coated fabrics via a non-solvent induced phase separation (phase inversion) method.

The aim of this paper was to introduce a novel, facile, low cost, and environmentally-friendly method to produce an APC coated fabric from a PA by solvent-casting of only one component, specifically, PA66 onto the PA66 fabric in order to have strong adhesion between the coating and the fabric as well as enhanced recyclability.

MATERIAL AND METHODS

Materials

The formic acid used in this work was supplied by Sigma-Aldrich (ACS reagent grade, >98%). The PA66 plain woven fabric was provided by FOV Fabrics AB (Borås, Sweden). As a polymer source to produce a solution, PA fiber production waste from the weaving process at FOV Fabrics was used.

Composite Preparation

All-polyamide composites were prepared in the form of a flat laminate on the substrate fabric by an isothermal immersionprecipitation method. Seven solutions of PA production waste in the formic acid were made by dissolving 17, 23, 29, 35, 41, 47, and 53 g of PA in 100 g (82 ml) formic acid at room temperature. In low concentrations (less than \approx 30% w/w), PA66 readily dissolves in the formic acid at room temperature, but for higher concentrations, the solution should be agitated for a longer time. In order to assure the completion of dissolution and have the same agitation condition for all the solutions, the sealed solution flasks were put in a shaker at the speed of 100 rpm for 20 h at 55°C to obtain a homogeneous solution (hereinafter, called dope). The dopes were cooled to room temperature, and after centrifugation for 10 minutes at the speed of $16,000 \times g$ to remove the bubbles, the dopes were casted on a PA fabric with the size of 18 \times 24 cm fixed on a glass with adhesive tape, using a universal film applicator with a thickness of 175 μ m. Once the casting process was done, after waiting 15, 30, and 45 s (three different times), the glass plate (carrying the fabric and a layer of the PA solution on top of it) was immersed in a distilled water coagulation bath at room temperature to induce polymer precipitation (phase separation). From then on, the waiting time, which is the time between applying the solution to the fabric and immersion in the coagulant, is called gelling time. After 1 h coagulation (in the water bath), the composites obtained were first washed three times with distilled water and then they were held under light press between two sheets of filter papers and dried at 55°C in a vacuum oven $(\approx 0.1 \text{ bar})$ for 2 h. The composites were named according to their corresponding solution concentrations and gelling time (Table I).

Characterization Methods

The tensile properties were evaluated in accordance with the standard method ISO 527.¹⁵ Dumbbell shaped test bodies, 75 mm long (with the width of 4 mm), were tested on a MTS 20/M tensile tester (MTS Systems Corporation, Eden Prairie, MN), fitted with a 5 kN load cell and a special grip for films, using a crosshead speed of 5 mm/min. The gauge length, pre-load force, and first approach speed were 0.5 N, 2 mm/min and 33 mm, respectively. The thickness of the composites was measured by Elastocon thickness meter (Elastocon AB, Sweden). A



 Table I. Solution Concentrations, Gelling Times, and Codes for APCs

Code	Solution concentration (%)	Gelling time (s)
APC1715	17	15
APC1730	17	30
APC1745	17	45
APC2315	23	15
APC2330	23	30
APC2345	23	45
APC2915	29	15
APC2930	29	30
APC2945	29	45
APC3515	35	15
APC3530	35	30
APC3545	35	45
APC4115	41	15
APC4130	41	30
APC4145	41	45
APC4715	47	15
APC4730	47	30
APC4745	47	45
APC5315	53	15
APC5330	53	30
APC5345	53	45

minimum of five test bodies was tested for each material. The specimens were all cut in the warp direction of the fabric.

To investigate the viscoelastic properties of the composites, dynamic mechanical thermal analysis (DMA Q800, TA Instruments, Waters LLC) was performed on the prepared composites. The specimens were run with a film tension clamp using the temperature ramp procedure with a sample dimension of $\sim 15 \times 9$ mm. The temperature ranged from room temperature to 180°C with a heating rate of 3°C/min; the frequency and the amplitude were 2 Hz and 15% strain, respectively.

Scanning electron microscopy (SEM) was used to monitor the fracture surface morphology of the cross-sections of the composites. The specimens were obtained by quenching in liquid nitrogen and breaking by hand. The studied surface was sputtered with a layer of gold before the measurements. SEM analysis was performed using AIS2100 (Seron Technology, Korea) operated at an acceleration voltage of 18 kV.

RESULTS AND DISCUSSION

To overcome the recyclability problem of the PA-coated fabric and to increase the adhesion between the coating and the fabric, a PA-coated PA fabric single-polymer composite out of PA66 was prepared. It was done by applying the PA66 solution to the PA fabric by means of a universal film applicator and consecutively, coagulation in a water bath as a non-solvent to induce phase separation (phase inversion). As a result, a composite composed of a thin continuous PA layer (the coating) and a PA textile fabric out of the most common type of aliphatic PA (PA66) was obtained. The hypothesis in this work was that since the coating and the fabric were the same, and there was a common solvent in between them, the adhesion would be very strong. Different properties were analyzed, and the process parameters were optimized.

Mechanical Properties

The maximum force needed to break the composite in the tensile mode, as well as elongation at max, and the thickness of the APCs are shown in Figure 1. As the thickness of the composites was not the same, the maximum force was chosen as the criterion to compare the mechanical properties of the composites. This was decided since the mechanical characteristics of the fabric (a spun fiber, high crystallinity), and the formed film (an amorphous film, low crystallinity) differs greatly with each other. Moreover, in different composites, different amounts of PA film exists; thus, in this case, the stress, that is, force divided by cross-sectional area, is not a good criterion to compare the strength of the APCs.

The results from the tensile testing giving the maximum force (Figure 1) showed a slightly good trend. The results indicated the increase of mechanical strength with the concentration of the dope for each gelling time group individually. If only the 15 s gelling time composites were considered, the maximum force needed to break the composites increased as the corresponding dopes contained more PA (higher concentrations). This trend was valid for 30 s and 45 s gelling times. However, if the three different gelling times of one single group of the dope concentration were considered, the trends were not the same for all the 7 groups. There is no clear trend in increasing the gelling time. Some dope concentration (for example 17%) possibly show an increasing trend of the maximum force when the gelling time is increased. However, the trend is not so significant considering the standard deviations.

Comparing the fabric maximum force (Figure 1) with the other APCs, showed that by applying PA dope on PA fabric, the fabric became weak in terms of mechanical strength that can be attributed to the dissolution of the surface of the fabric by the solvent existing in the dope. However, the 53% APCs had very close maximum force values to those of the fabric. This decreasing in mechanical properties can be regarded acceptable in compensation of giving a new property to the fabric (making the fabric gas-/water-proof) and resulting a coated fabric. For elongation at max, more or less the same trend was valid with the exception that the APCs made out of concentrated PA dopes (especially 47% and 53%) had higher elongation at max values. This was due to the difference between elastic properties of amorphous and crystalline polymers. The fabric is made from a spun polymer fiber that has a higher crystallinity than the formed-film that is an amorphous polymer. Therefore, by increasing the amorphous (more elastic) polymer in the composite, it is reasonable to have a higher elasticity, elongation at max value. It is in accordance with the rule of mixtures mechanistic model,²⁷ which says the resulting mechanical properties of a composite, is the individual property of the components based on their volume fraction.



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Figure 1. Max force, elongation at max and thickness in the tensile test of the APCs as well as the fabric, versus different solution concentrations for 15 s, 30 s, and 45 s gelling times. The variations can be attributed to many factors, mainly the dissolution power of the dopes. "Fab" stands for fabric, and it has been added to the graph for comparison purposes.

By increasing the PA concentration of the dope solution, the content of formic acid in the solution with respect to the polymer, that is, the ability of the dope to dissolve the surface of the fabric decreases while the amount of the PA film on the fabric increases. The more the fabric surface dissolves, the more the strength of the fabric diminishes. On the other hand, in order to obtain a good adhesion of the PA layer to the fabric substrate, it is necessary to have a certain amount of surface dissolution. It is obvious that for higher concentrations of the dope, the composite has a higher maximum force and is thus mechanically stronger. It is due to the higher concentration of the film formed (higher loading of PA) on the fabric and better opportunities to form entanglements between the formed film's polymer chains and the polymer chains of the fibers in the fabric. The mentioned trend is slightly valid when increasing the gelling time for each dope concentration. By increasing the penetration time for the solvent into the fabric surface (and dissolving the surface of it), logically the amount of penetrated solvent (and hence the number of polymer chains that can entangle with the chains of the polymers in the film) increases; while the concentration of the solvent is the same for the three different gelling times.

The thickness of the composites (Figure 1) varied with different concentrations of the dopes and even with various gelling times. It can be explained as follows: by increasing either the dissolution power (lowering concentration) of the solvent or the dissolution time of the solvent in the dope to dissolve the fabric (gelling time), a greater amount of the fabric is dissolved; hence, the thickness decreases. The trend is more or less valid for all the composites except for APC2945, which can be related to a critical concentration or experimental errors. Although the same universal film applicator (with the thickness of 175 μ m) was used for all of the composites, the thickness varied. The thickness decreasing with the gelling time indicated that the time dedicated to the solvent to dissolve and go through to the fabric, had an inverse proportion with the thickness due to the dissolution power, as mentioned before. The increasing of the thickness by increasing the dope concentration may very well be attributed to increased polymer content in the formed-film. A film with more polymer content would more than likely have an increased thickness. However, the decreasing the thickness in each group by increasing the gelling time, supported the "dissolution power" proposal for the thickness variation.

We propose that entanglement effects may explain the trend in the maximum force and elongation data shown in Figure 1. At low polymer solution concentrations, there are fewer entanglements so that the polymer can more easily order its chains.²⁸ These ordered regions can serve as nucleation sites, promoting more crystallization in the precipitated sample.²⁸ If the polymer entanglement proposal is correct, the increasing elongation at max by increasing the dope concentration is due to having a higher amount of amorphous regions at the higher concentrations of the dope. Therefore, they are more elastic than those at the lower concentrations. At higher dope concentrations, however, polymer entanglements can interfere with the ordering (making the ordering difficult) and thus reduce the number of ordered formed regions.²⁸

If the polymer entanglement proposal is correct, then there is a critical concentration, C^* , at which entanglements begin to



Figure 2. An exemplary DMTA graph for APC4745 composite; maximums, onset-, mid-, and end-points, as well as step transition, are shown. The straight lines are drawn by the DMTA machine's software to find the points shown by arrows.

form, and the solution becomes semi-diluted.^{29,30} This critical concentration can be approximated as follows³⁰:

$$C * = l/[\eta]$$

where $[\eta]$ is the intrinsic viscosity. Intrinsic viscosity $[\eta]$ is related to the molecular weight (*M*) by the Mark-Houwink equation, defined as:

$$[\eta] = KM^a$$

where *K* and *a* are constants, that depend on the polymer, solvent, and temperature.²⁸ *K* depends on the width of the molecular weight distribution, and *a* is a measure of the polymer-solvent interactions.³¹ Thus, by increasing the molecular weight of PA, C^* will decrease, and entanglements will occur at lower concentrations. This means that every time that PA scraps (as PA source in this work) with a different molecular weight is going to be used, the parameters need to be optimized again.

Another reason for improved mechanical properties by increasing the dope concentrations is probably due to increased mass (surface weight). Higher dope concentrations contain a higher amount of polymer and form a denser layer on the fabric. At very low dope concentrations (less than 35%), the concentration of the solvent is so high that the dissolution of the fabric is too much and more or less destroys the fabric according to maximum force results. But at higher concentrations, especially at 47% and 53%, the amount of the solvent seems to be optimized so it does not dissolve the fabric more than what is needed; i.e., dissolves the fabric's surface only to an extent that is required to establish the adhesion. Therefore, the higher concentrations (41%, 47%, and 53%) are more optimized concentrations to prepare the APC in terms of the mechanical properties. With the same argument, the longer gelling times in each of the aforementioned three optimized concentrations give better mechanical properties to the corresponding composites.

Viscoelastic Properties

The three composites APC1715, APC1730, and APC 1745 were yielded during the analysis in the DMTA machine at 56°C, 43°C, and 32°C, respectively; therefore, no results were reported

for them. An exemplary DMTA graph for the APC4745 composite with appointed maximums, onset-, mid- and end-points, as well as step transition, are shown in Figure 2.

Although there is no regular trend for the peak of the loss modulus (\approx glass transition temperature, T_g) of the composites, as for the peak of the loss modulus versus temperature curve (Figure 3), it is clear that APC4130 has the highest value. For the two more concentrated dopes, i.e., 47 and 53%, loss modulus



Figure 3. Loss modulus, tan delta, and stiffness of the APCs versus different solution concentrations for 15 s, 30 s, and 45 s gelling times.



for 30 s gelling time is greater than for 15 s and 45 s. However, for the rest dopes, it is smaller than the other two gelling time. The physical and mechanical properties of the coating, along with its glass transition temperature and its thickness are key



parameters affecting properties, such as the interfacial strength and the fracture toughness.³² Loss modulus describes the viscous properties, meaning that composites with longer gelling time have better elastic properties than those with shorter gelling time. The variations in the viscoelastic results (Figure 3) are probably related to a larger part of the PA fabric being dissolved and changing it from a higher crystalline form (spun fiber) to a more amorphous polymer, from lower to higher dope concentrations as well as from shorter to longer gelling times. Since amorphous polymers are disorganized, they can align in the direction of the force imposed on them, and by this, they can dampen the energy, at least more than the amount of dampening energy in more crystalline regions.

By increasing the temperature, the stiffness of the polymer decreases. The temperatures at which the drop in stiffness occurs are shown in Figure 3. Comparing the stiffness step transition onset point and gelling time of the APCs at various dope concentration, it is difficult to infer the effect of the gelling time/dope concentration on the stiffness. Again there is no clear and regular trend, but longer gelling time composites have higher storage moduli (see Figure 4). When the gelling time is longer, the solvent has more time to penetrate into the fabric, resulting in better adhesion. This indicates that the polymer chains in the formed film and the polymer chains in the dissolved parts of the fabric can form more entanglements with each other. Therefore, the longer gelling time gives not only better adhesion, but also improved damping properties that can dissipate more energy, which, as a result, gives higher tan delta (loss tangent) (Figure 4). In this system, there might be a critical entanglement degree in which the polymer chains can entangle in each other enough to make the resulting composite stiff, but not to the extent that make it rigid. The variations, especially in 35% and 41% dope concentrations, can be due to the critical degree of entanglements. As Varelidis et al.33 discuss, the glass transition temperature and the thickness of the flexible interlayer have been identified as the major parameters affecting the end properties of the composite. Therefore, in this regard, APC4130 is the optimum APC.

Morphological Properties

According to the scanning electron microscopic images of the cross-section (Figure 5), in almost all the APC specimens there is a good adhesion between the formed film and the fabric. In other words, the boundaries between the fiber of the fabric and the formed film are not clear because they have faded due to the adhesion of the two components.

Figure 4. Tan delta, storage modulus, and loss modulus for some selected All-Polyamide Composite (APC) coated fabrics. Although there is no clear and regular trend, longer gelling time composites have higher storage moduli. Longer gelling time gives more opportunity for the solvent to penetrate into the fabric, resulting in better adhesion and more entanglements, which eventually improved damping properties that can dissipate more energy, which as a consequence gives higher tan delta. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary. com.]



Figure 5. Cross-sectional scanning electron microscopic images of the APCs. From top to bottom, 17%, 23%, 29%, 35%, 41%, 47%, and 53% polyamide dope concentrations, and from left to right 15 s, 30 s, and 45 s gelling times, respectively. The micrographs of the specimens were taken after quenching them in liquid nitrogen and breaking by hand. The magnification is 500 times for all of the micrographs.

At higher concentrations of the solvent (lower concentrations of the polymer solution), the dissolving power of the dope is higher; hence, it can penetrate more into the fabric and dissolve a larger part of the fabric, e.g., shown in APC2930 and APC2945 SEM micrographs. It is obvious that a larger part of the cross-sectional area of the fabric is dissolved, and the



Figure 6. Illustration of the porosity in APC5345 with different magnifications.

polymer chains are inter-diffused into each other, compared to the SEM micrographs for APC5330 and APC5345. Although higher surface dissolution helps to create a better adhesion between the fabric and the formed film, it also degrades the fabric structure and changes the fabric from a fibrous form to a film form. Fibers are spun polymers and pose a high crystallinity and thus a good strength while films are amorphous. Therefore, converting PA from a high crystalline form to a less crystalline (high amorphous) form is not favorable from the mechanical point of view. The amount of the surface dissolution should be as low as possible to impart a good adhesion between the fabric and the formed film, but higher dissolution is not favorable.

In the APC5345 composite, good adhesion is seen, but there are some voids in the formed film (Figure 6). The voids make the film porous, which can be due to the difficulty of the solvent inside the dope to evaporate from the gel during the coagulation process due to the high viscosity of the dope. When the solvent migrates from the gel, as the dope concentration/viscosity and polymer chains entanglements are great while leaving,



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the solvent leaves the channel and creates a void. Because of the entanglement, the chains next to the migrating solvent molecules are not able to rearrange themselves to fill the channel formed by the solvent while leaving. In other words, the solvent creates a route for itself to be able to come out and by this, leaves behind a channel. This property can be useful for some applications, in which the porosity of the composite is required, like supported membranes.

CONCLUSIONS

The preparation of a novel all-polyamide composite (APC) coated fabric, a specific form of single-polymer composites, made through a phase inversion method is described in this report. The prepared composite has a strong adhesion between the two constituents due to the use of the same polymer (PA66) in the constituents and a common solvent. The composite is fully recyclable since it contains no other materials except PA, which can be melted or dissolved and reused like a virgin PA source. The optimum concentration is 47% if a dense film is needed, but if it is necessary to present a porous film on the fabric, then 53% solution should be used to prepare the APC membrane, that can be regarded as a fiber-supported membrane. The composite can be tailored in various ways with different properties depending on the end-use requirements. The prepared composite has a wide range of semi-structural applications to provide lightweight, architecturally striking solutions as well as wide-span surfaces, membrane-cable structures, hanging roofs, pneumatic constructions, water-/gas-proof textile reactors, temporary houses and tents, facade coverings, container linings, and tarpaulins. Apart from the novelty of the method to prepare a single-polymer composite coated fabric out of PA, in this work, a new method was introduced to convert a low value-added waste (PA scraps) to a high value-added product (APC coated fabric).

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