Study of Effect Ammonium Persulfate and Amino-Methyl-propanol in grafting Maleic Anhydride on Polietilene in the Making Softener.

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Abstract : One of the problems associated with this grafting process is the formation of lumps and granules. To overcome this problem, ammonium persulphate (APS) as catalyst together with amino- metil- propanol (AMP) having 95% purity as neutralizing agent will be used in grafting 25% MA solution with 25% PE solution. To overcome this problem, ammonium persulphate (APS) as catalyst together with the amino-methyl-propanol (AMP) having 95% purity as neutralizing agent. Characterization of the synthesized product includes chemical , bonds identification using Fourier Transform Infra-Red (FTIR) spectroscopy, viscosity, solid content and softness. The information obtained from FTIR spectroscopy shows that C-O bonds have been replaced by C-N, C=O, C-O bonds and that previously absent N-H bonds are formed. As a whole , this study also shows that PE-g-MA 9% synthesized at 95^{o}C is the most stable solution without the formation of lumps nor separation upon cooling.

Keywords: PE 25% *solution* , MA 25% *solution* , PE-g-MA Key word: PE 25% *solution,* 25% MA *solution,* PE-g-MA

1. INTRODUCTION

In this research trials to overcome the problems associated with the quality of the resulting polymer product, through a synthesis between the material of PE with MA. Proses sintesis tersebut menggunakan bahan katalis, yaitu *ammonium persulfate* (APS) The synthesis process using a catalyst material, namely *ammonium persulfate* (APS) and neutralizing the *2-amino, 2-methyl 0.1-propanol* (AMP) is formed so that the transplant MA in PE (PE-g-MA) are homogeneous. This process is expected to be more effective and economical in terms of time and cost.

Role of softener in the textile industry are used as binding material in the fibers of the fabric, so the stiff fabric becomes softer and smoother surface. Results of synthesis of PE-g-MA depends on composition of the catalyst, and a neutralizing composition. The emphasis of this research is to determine the composition of the MA to produce the compound of PE-g-MA which has a pH close to stable alkaline or pH 5 to 6, there is no clumping and no grain resulting in PE-g-MA.

2. MATERIALS AND METHODS

MA grafting on PE using polietilene 25% *solution, Maleic Anhydride* 25% *solution, demineralized water* (distilled water), *ammonium persulfate, 2 - amino, 2-methyl, 1 - propanol* 95%. MA in PE grafting process carried out by maintaining the *solution* obtained does not form clots, as well as observing color changes occur, changes in bonding with *Fourier transform infrared spectrophotometer-red* (FTIR), as well as the physical characteristics of the *dry film* was smooth, without granules. To generate a PE-g-MA optimal, so in this research, a characterization that includes the degree of acidity of the solution, solution viscosity, solid content of the solution, particle size of the solution, changes in bonding, and tenderness materials

2.1. Goal Research.

1. Optimization of the grafting of PE with MA to replace conventional materials which are widely used surfactant. To eliminate clumping and grains conducted variation MA 95 concentration of PE is the use of surfactants will be generated composition of PE-g- MA is not caking

2. Characterize the quality of PE-g-MA before and after the addition of AMP in 1995. PE-g-MA produced were identified based on morphology (type of bond) PE-g-MA produced by spectrophotometer *Fourier transform infra-red* (FTIR), measurement of the degree of acidity, solid content, particle size, viscosity, and softness of the material, before and AMP 95 after the addition.

2.2. Research Contribution.

MA grafting on PE 25% *solution* 25% *solution* made in this study is expected to be an innovation that could replace the softener softener that can overcome the conventional coagulation and sedimentation.

2.3. Polyethylene.

With increasing temperature, the bond will be easy experienced polietilene crosslinking *(crosslinking)*, because the stronger the PE energy. Application of heat to the PE, making the molecular chains of PE is relatively easy to move and easier to friction with the molecules - the other molecules. PE material experiencing the crosslinking can be easily processed, and the basic structure of PE has a better strength.

2.4. Introduction of Softener.

Softener in this research is a chemical that is used for textiles, fabrics, clothing, etc., which can alter the surface of the fabric becomes softer and softer to the touch, and characterized as silk. Softener used to improve fabrics, knitted fabrics, fiber, yarn, fabric curtains, as lubricating oil cloth, and softener surface. Therefore, the softener incorporated into the final forming of a fabric. Standard general parameters are conducted to determine the quality of the softener include: [1]

1. Coefficient of friction ;

   fiber lubricant can be used to reduce the coefficient of friction and make the fabric feel softer.

2. Viscosity;

   viscosity materials ranging softener such as liquid (lubricating oil) until semi-solid *waxes.* Low viscosity indicates that the softener have a fairly good quality because it can make the fabric feel softer.

3. Color;

   Results from the softener does not destroy or produce a color change in the ingredients. Softener color ranges from translucent white to yellowish white.

4. Odor;

   The smell generated from the softener does not affect changes in the structure of the basic ingredients of fabric.

2.5. Maleic Anhydride. Maleic Anhydride (MA) is the main ingredient used in transplantation PE to produce PE-g-MA. MA has a high elektronegatifitas clusters and contains a lot of 'O' (oxygen), so it has quite polar nature. MA has a sensitive nature, flammable and will experience irritation when the subject's hand. MA has a boiling point of 200 ^{o} C. [2]

MA has the properties of soluble *(solubility)* at temperatures below <50 $^\circ$ C. The MA has a molecular weight of 98.06 g / mol with a melting temperature range between 51 ^{o} C to 53 ^{o} C

2.6. Grafting of Maleic Anhydride In Polietilene (PE-g-MA)

In this research, MA grafting on PE in liquid form , which serves to reduce the clotting solution. In this case the Supreme Court established in 25% solution dicangkokan on PE in the form of 25% APS solution with the addition of catalyst. APS is not alter the structure of PE-g-MA. Comparison of PE-g-MA is expected that less than 1: 3 [3], as shown in Figure 1. Catalyst that enables the radical reaction of PE as the backbone in transplant MA. [4]

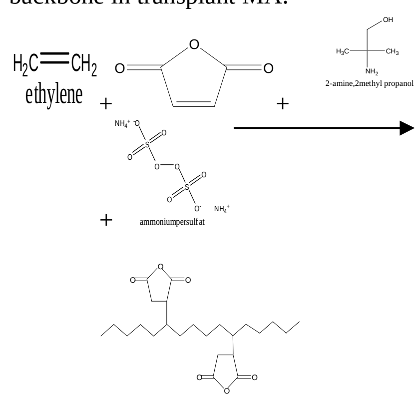

Figure 1. The process of transplantation MA in PE with the addition of APS and AMP

2.7. Materials

1.Polietilene 25% solution. Polietilene 25% solution or in a commercial language is polietilene **ceranine** is one which is available on the market and usually in the form of wax polietilene. PE 25% solution has a solution containing 25% and 75% liquid consisting of inbentin T-10, AMP 95 and distilled water.

2. Maleic Anhydride 25% solution. The product will be used in this research that is MA 25% *solution*, which is prepared by mixing ekaline ie there were 38 grams, 42 grams inbentin, MA as much as 500 grams and 1,500 grams of distilled water (1500 cc) with a total of 25% *solution* MA 2080 gram. This was done so that the PE-g-MA that will be produced as stable as the absence of coagulation and condensation separation does not occur, and the results of the *dry film* looks more gentle. In this case the softness of the film with a *dry film* is the main thing in the marketing of products softener.

3. Ammonium persulfate Ammonium persulfate (APS) has the structure of $(NH_{4)2}S_2O_8$ is an intermediate solution as a strong oxidation. APS will be very soluble in cold water. Very large temperature changes, the APS solution will be approached. Besides as a powerful catalyst, APS is also a radical initiator.

4. Amino-Methyl-propanol In this study, using AMP species that have a purity of 95% or often called the AMP 95. AMP 95 amino resulting in increased bonding functional, more flexible and more results than the bond of other amines. Besides AMP also functions as a neutralizing.

3. CHARACTERIZATION AND TESTING MA grafting on PE 25% *solution* 25% *solution* made by *Fourier transformation* pengkarakterisasi *Infra-red* (FTIR) and conducted karakteristisasi degree of acidity, solid content, viscosity, the softness of the material, particle size so that can know the results of MA grafting on PE are stable.

3.1. The materials are prepared:

a. PE 25% *solution* = 1000 g

b. MA *solution* 25% =10,30,50,70,90 gr

c. Ammonium persulfate(APS)=5 gr

d. *Demineralized water* (DW) 10 cc

e. 2-amino, 2-metil,1-propanol =25 gram

3.2. Flow of work based on composition, time and temperature

Figure 2 represents the flow of work based on composition, time and temperature are carried out in experiments, with the work as follows:

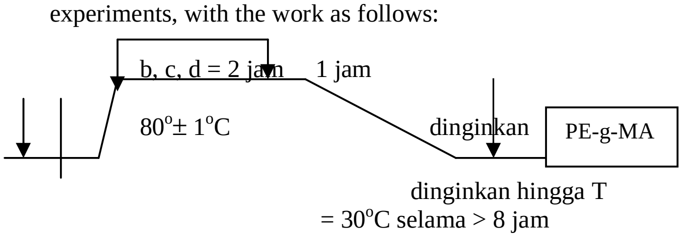

Figure 2. Flowchart of making PE-g-MA based on composition, time and temperature

4. RESULTS AND DISCUSSION

Then research from the solid content of PE-g-MA, the degree of acidity of PE-g-MA, viscosity of PE-g-MA, the particle size of PE-g-MA , changes in the bond of PE-g-MA, and the softness of PE-g-MA. These data can be analyzed as follows:

4.1. Analysis of Solid Solution

Can see the amount of solid content of PE-g-MA before the addition of AMP 95 given declining. When the temperature is increased the solid content would be higher because the bonding between atoms C-H-O bigger. This causes the amount of H_2O vapor is very small. The more the Supreme Court granted the 25% *solution* of PE grafting, the concentration of H_2O evaporates more and more. More and more 25% MA grafted on the PE *solution*, solid content decreases

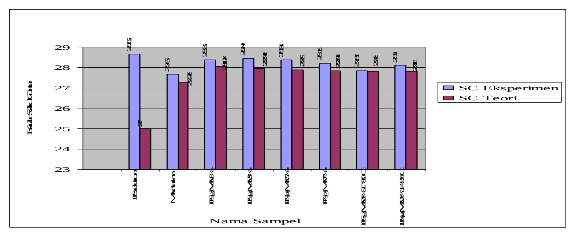

Fig 4.1.1. Graph solid content of PE-g-MA before the addition of AMP 95

Solid content due to differences in the grafting of PE with MA solution formed consisting inbentin T-10, ekaline F and have the nature of the catalyst, which makes reaction even more solid and transplantation of H_2O content of less and less, so the smaller the value of solid content.

The difference in solid content of PE-g-MA experimentally and theoretically very large. This was due to strong carbon bonds so that after the addition of AMP 95 to form solid bonds. In addition the study was not conducted AMP 95 in PE-g-MA 1%, this is because the pH in the PE-g-MA 1% had reached pH 5.69.

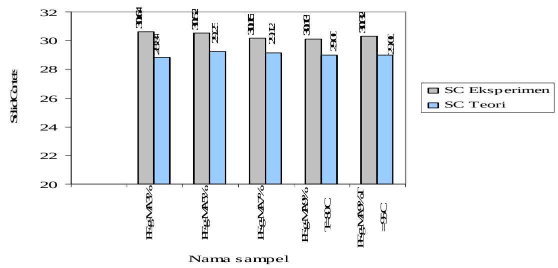

Fig. 4.1.2. Graph solid content of PE-g-MA after the addition of AMP 95

In the analysis of solid content can be stated that the PE-g-MA 9% at a temperature of 80 ^{o} C is the optimal composition, this is because the solid content MA which grafting PE is a large. Comparison of experimental solid content with very little theory is 0.05, in this case because the amount of H_2O concentration on fewer and the number of transplants that evaporates more.

4.2. Analysis of Degree of Solvent Acidity

Analysis of degree of acidity (pH) of PE-g-MA with MA 25% solution neutralized with AMP. Can be seen that by increasing the amount of PE grafted with MA, the lower the pH value. This is because the pH value which is very acidic and the nature of the MA-containing inbentin T-10 ekaline F and MA, which makes more acid.

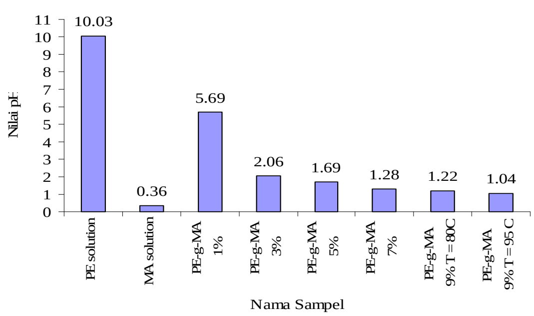

Fig.4.2.1. Graph value acidity analysis of PE-g-MA before the addition of AMP 95

Based on the analysis of degree of acidity, then the PE-g-MA 1% represents an optimal composition for acidic pH values are not valuable. Toward alkaline pH caused the formation of MA with the PE ratio is very much that is 1: 100. The rate difference resulted in PE and MA grafted PE is still difficult. Thus the acidity of the analysis can not be concluded PE-g-MA is good, because to change the pH becomes alkaline requiring neutralizing (AMP), which phones. But when viewed from the acidity value, the value of PE-g-MA 9% is good, because the acidity is very little value. The pH of PE-g-MA is small ie 1.91 and 1.67 was due to PE (pH PE = 10:03) which has undergone a transplant MA (MA pH = 0.36) is good.

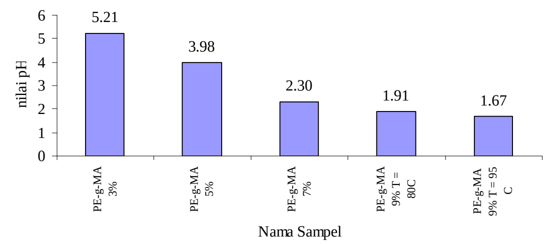

Fig. 4.2.2 Graph acidity analysis of PE-g-MA after the addition of AMP 95

4.3. Analysis of Viscosity Solutions

PE-g-MA 1% have to use the mixer 4, this is because the PE-g-MA produces a very strong character and is a solution which approximates the pH alkaline, OH binding solution $^{-}$ to avoid an excess of H $^{+}$ to make the product more viscous. This caused higher levels of OH $^{-}$ then produce bigger products have a corrosive, thus making the smaller of the inhibition. But the higher the temperature to be done, the greater the speed, because the PE-g-MA 9% at a temperature of 95 ^{o} C, a very dilute solution. In the PE-g-MA 9% with a temperature of 80 ^{o} C increased the viscosity values, this is because the transplant has resulted in a stable solution.

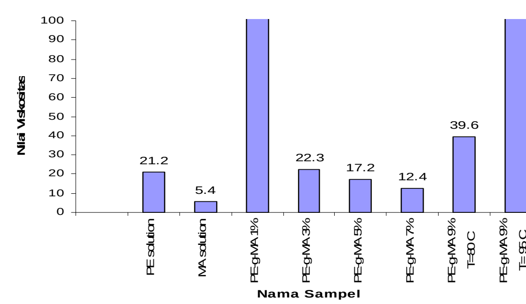

Fig. 4.3.2 Graph viscosity analysis of PE-g-MA before the addition of AMP 95

In the analysis of viscosity can be concluded that the PE-g-MA 9% at 80^{o}C is very weak, even at a temperature of 95 ^{0} C can use the rotating speed 10 rpm for being too thin. This is what is expected in the industry. PE-g-MA is diluted, because the stability of the solution. Stability of the solution resulted in reduction of production costs.

4.4. Particle Size.

PE-g-MA after the addition of AMP in 95 does not look big difference with PE-g-MA before the addition of AMP in 95. Seen PE-g-MA 3% and 5% have a very large particle size, this can also be seen on the characterization by FTIR.Namely the change in bond grafting of MA on PE invisibility of the two peaks in the PE-g-MA 5% and 7%, resulting in a solution of very large separation during cooling.

Table 4.4 Results of particle size analysis of PE-g-MA before the addition of AMP 95

Nama Sampel	Diameter	Ketebalan	Volume
	nm	nm	%
PE solution	25.15	8.441	100
MA solution	12.33	1.936	100
PE-g-MA 1%	667	84.92	100
PE-g-MA 3%	∞	∞	∞
PE-g-MA 5%	∞	∞	∞
PE-g-MA 7%	2001	207.5	100
PE-g-MA 9% T=80 C	603	70.38	100
PE-g-MA 9% T=95 C	80.21	8.975	100

From particle size analysis shows the PE-g-MA has a very large size approaching the size of the micro. This solution caused the separation of PE-g-MA after cooling, except on PE-g-MA 9% at a temperature of 80^{o}C, even at a temperature of 95^{o}C shows a value approaching nanometers.

Table 4.4 Results of particle size analysis of PE-g-MA after the addition of AMP 95

Nama Sampel	Diameter	Ketebalan	Volume	kadar AMP
	nm	nm	%	Gram
PE-g-MA 3%	1728	179	100	14.77
PE-g-MA 5%	∞	∞	∞	25
PE-g-MA 7%	∞	∞	∞	25
PE-g-MA 9% T=80 C	829.4	86.04	100	25
PE-g-MA 9% T=95 C	647	80.63	100	25

4.5. Change Bond.

By analyzing changes in the FTIR bond, then the graft reaction that occurs between the PE *solution* with 25% MA 25% *solution* is often called PE-g-MA with a catalyst AMP and neutralizing APS has been successfully performed. In addition to analyzing the degree of acidity, viscosity, particle size, solid content, a chemical also made observations on grafting reaction products was done by observation of functional groups in these compounds using infrared spectroscopy FTIR types. A blend of PE-g-MA before the addition of AMP in 1995 showed that the intensity of the wave ±1090 cm^{-1}, PE 25% *solution* containing C-O bonds, together with the PE-g-MA 1%, but after the PE-g-MA has succeeded in grafting MA on PE, which is turned into a C-N bond. In the intensity of 1500-1750 cm $^{-1}$ waves, visible changes in bonding at the PE-g-MA, namely the change of the C-C bonds on the MA to C = O bond in PE-g-MA. In the wave intensity ≈ 2000-2400 cm $^{-1}$, not seen the peak in the PE, but after transplantation, then on the analysis of PE-g-MA on the top of the results of FTIR looks MA grafting on PE namely N-H bond.

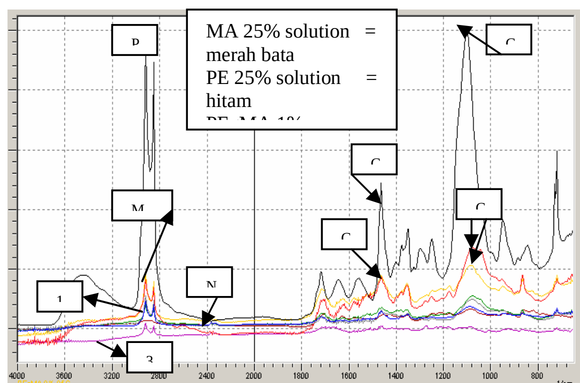

Figure 4.5.1. Changes in bond PE-g-MA before the addition of AMP 95

AMP 95, on the PE-g-MA 3% and 5% with the intensity of the wave ± 1090 cm $^{-1}$ has two peaks with the CO bond, whereas the PE-g-MA 7% and 9 % visible change in bonding of C-O to C-N. So also on the intensity of the wave ≈ 2000 - 2400 cm $^{-1}$ peak changes, which are not visible from the PE peaks become visible peaks and produce N-H bond,

Changes in bonding also occurs in the PE-g-MA 9% both at 95^{o}C before and after the addition of AMP 95 there was a change from the anhydrides bond (C = 0) to bond aldehides (C ≅ O) on the wave intensity ≈ 1500 to 1750 cm $^{-1.}$ That caused by neutralizing 95

AMP produces H_2O bond becomes more so the triple bond.

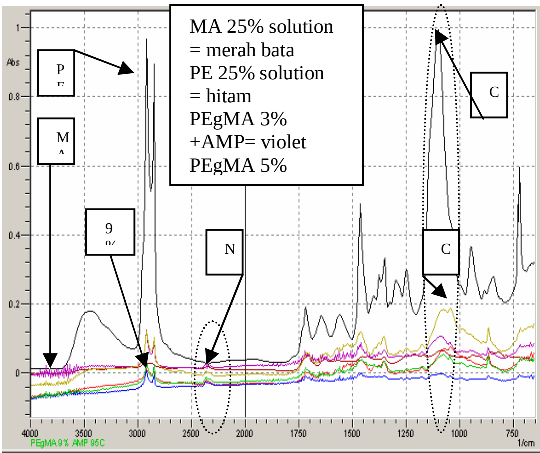

Figure 4.5.2. Changes in bond PE-g-MA after the addition of AMP 95

PE-g-MA 9% at a temperature of 80 ^{0} C and 95 ^{0} C both before and after the addition of AMP 95 combined, it will be seen more clearly the difference wave intensity Δpeak at 1500-1750 cm $^{-1,}$ ie a change from C = O bond O ≅ C to bond

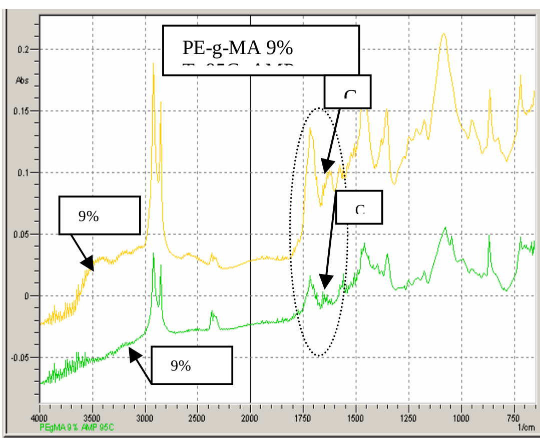

Figure 4.5.3. Changes in bond PE-g-MA 9% before and after the addition of AMP in 95 at a temperature of 95 ^{o} C

The result of FTIR analysis of the changes seen bonding with PE-g-MA which is amended bond in PE-g-MA 9% with a temperature of 80 ^{0} C, even at a temperature of 95 ^{0} C there is a change to a triple bond, C ≅H at wavelength 1500 - 1750 cm $^{-1.}$ In addition to visible changes in the structure of CO CN at a wavelength of ±1090 cm $^{-1}$ in MA 7% and 9%, even at a wavelength of ± 2000 cm $^{-1}$ seen in the NH bond of PE-g-MA.

4.6. Softness Materials.

One of the most important factor in the desired outcome is the softness of the material, namely the absence of lumps and grains. Softness of PE-g-MA was observed in *Dry Dry film* will show a very different *movie*. *Dry film* looks at the PE-g-MA 9% at a temperature of 80 ^{o} C is more subtle almost no grain.

Fig. 6. Separation of PE-g-MA after cooling

Dry film forms on the PE-g-MA AMP after the addition of 95 more shows a more stable form of the PE-g-MA 9% at a temperature of 80 ^{o} C and at a temperature of 95 ^{o} C.

From the analysis of the softness of the material with *dry film* can be seen that PE-g-MA 9% at a temperature of 80 ^{o} C occurred without the existence of subtle forms of coagulation, even at a temperature of 95 ^{o} C, the film looks more smooth with a brighter color

5. CONCLUSION.

This study has been carried out the transplant process MA 25% *solution* at 25% PE with catalyst *solution* Ammonium Persulfate (APS) and Amino-Metil- Propanol (AMP) . Characterization of the grafting process includes measurement of the degree of acidity with a *pH of metro Ωtools*, measurement of *nanometer* particles by means *zetasizer*, solid content and viscosity with a viscometer, the softness of the material with *the film* and the *dry* method changes in bonding with *FTIR* spectroscopy. Concentration MA in this study varied by 1, 3, 5, 7, and 9% at a temperature of 80 ^{o} C, and the MA of 9% at a temperature of 95 ^{o} C for comparison. The experiment shows that PE-g-MA 9% at a temperature of 80 ^{o} C is the most stable composition. Increasing the temperature to 95 ^{o} C on the composition gives a better result that is not found during testing of clotting material softness.

Acknowledgements

For further research on MA grafting on PE, the catalyst can be used in addition to AMP in 1995 that could increase the pH value is more significant. With a higher pH value would increase the storage life of materials.

The process of manufacture / synthesis of PE-g-MA can be done at higher process temperatures from 95oC, but lower than the melting point of PE (103-108 ^{o} C) to determine the saturation value of PE-g-MA.

Should be also investigated using the *Gas Mass Cromatografi Spectrometri* (GCMS) to better determine the change of bonding at the PE-g-MA

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