



Enhancing Paint Performance Through the Incorporation of Thermoplastic Acrylates With Varying Proportions of a Sustainable, Bio-Based Gum Rosin Acrylic Monomer (GRA)

Sürdürülebilir, Biyo-Esaslı Sakız Reçinesi Akrilik Monomerinin (GRA) Değişen Oranlarda Termoplastik Akrilatlarla Birleştirilmesi Yoluyla Boya Performansının Artırılması

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Abstract

In this study, a bio-based acrylic monomer (GRA) was synthesized through the acrylation of bio-based gum rosin. This monomer was subsequently employed in the synthesis of thermoplastic 1K (one component) acrylate polymers. These polymers are applicable in varnish and paint formulations, offering superior coating features, including increased gloss, enhanced adhesion, improved visual appeal, and robust impact resistance. The synthesized GRA served as a bio-based monomer in thermoplastic acrylate compositions at ratios of 2.5%, 5.0%, and 10.0%. The synthesized monomer and its resultant polymers were studied using gel permeation chromatography (GPC) and Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR). The thermal characteristics of the polymers were analyzed using a differential scanning calorimeter (DSC). The coating properties, including adhesion, gloss, hardness, drying times, and impact resistance, were evaluated and studied. The results indicate that increasing the quantity of gum rosin monomers in the polymers reduced the glass transition temperatures from 30.18 °C to 24.12 °C, which enhanced impact resistance and improved adhesion to metal surfaces. The adhesion is nearly doubled on metallic surfaces. Paints formulated with increased amounts of bio-based gum rosin exhibit exceptionally high gloss values, rapid drying times, and superior aesthetics compared to standard formulations. In comparison to the binders employed in currently available paints, the polymer containing gum rosin acrylate monomers can be utilized to manufacture paints and varnishes with enhanced coating capabilities. By the way, the paint formulations can be prepared without any addition of plasticizer.

Keywords: 1K acrylic, gum rosin, paints, thermoplastic acrylates.

Öz

Bu çalışmada, biyo esaslı sakız reçinesinin akrilasyonu yoluyla biyo esaslı bir akrilik monomer (GRA) sentezlendi. Bu monomer daha sonra termoplastik 1K (tek bileşenli) akrilat polimerlerinin sentezinde kullanıldı. Bu polimerler vernik ve boya formülasyonlarında uygulanabilir; daha fazla parlaklık, daha iyi yapışma, daha iyi görsel çekicilik ve sağlam darbe direnci gibi üstün kaplama özellikleri sunar. Sentezlenen GRA, termoplastik akrilat bileşimlerinde %2.5, %5.0 ve %10.0 oranlarında biyo esaslı monomer olarak eklendi. Sentezlenen monomer ve bunun sonucunda elde edilen polimerler, jel geçirgenlik kromatografisi (GPC) ve Yansıma-Fourier Dönüşüm Kızılötesi (ATR-FTIR) spektroskopisi kullanılarak incelenmiştir.

Polimerlerin termal özellikleri, diferansiyel taramalı kalorimetre (DSC) kullanılarak analiz edildi. Yapışma, parlaklık, sertlik, kuruma süreleri ve darbe direnci dahil olmak üzere kaplama özellikleri değerlendirildi ve incelendi. Sonuçlar, polimerlerdeki sakız reçinesi

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monomerlerinin miktarının artırılmasının, cam geçiş sıcaklıklarını 30.18 °C'den 24.12 °C'ye düşürdüğünü, bunun da darbe direncini artırdığını ve metal yüzeylere yapışmayı iyileştirdiğini göstermektedir. Metal yüzeylerde yapışma neredeyse iki katına çıkar. Artan miktarlarda biyo esaslı sakız reçinesi ile formüle edilen boyalar, standart formülasyonlarla karşılaştırıldığında olağanüstü yüksek parlaklık değerleri, hızlı kuruma süreleri ve üstün estetik sergiledi. Halihazırda mevcut boyalarda kullanılan bağlayıcılarla karşılaştırıldığında, sakız reçinesi akrilat monomerleri içeren polimer, geliştirilmiş kaplama kapasitesine sahip boyalar ve vernikler üretmek için kullanılabilir. Bu arada boya formülasyonları herhangi bir plastikleştirici ilave edilmeden hazırlanabilmektedir.

Anahtar Kelimeler: 1K akrilik, boya, sakız reçinesi, termoplastik akrilik.

1. Introduction

The development of innovative materials with enhanced properties relative to commercial counterparts advances trade and enhances the quality of life for consumers across all branches of chemistry. A wide range of polymers has been synthesized by including novel functional groups or compounds that improve their characteristics (Galus et al. 2020, Gowthaman et al. 2021, Mahmud et al. 2021, Petrunin 2022, Rehan and Usman 2023). Prominent trends in polymeric coatings include the development of eco-friendly coatings, the improvement of existing coatings' functionality, and the advancement of smart coatings with multifunctional properties (Yebra et al. 2004, Callow and Callow 2011, Faccini et al. 2021, Kumar et al. 2023). As global oil resources diminish and the production costs of petroleum-derived polymers escalate, along with their detrimental effects on environmental and human health, there is an increasing interest in alternative sustainable raw materials. However, the chemical diversity of renewable resources necessitates the resolution of several difficulties in the field of bio-based polymer materials. A primary concern is the design and production of bio-based polymer materials that have mechanical properties equal to or superior to those of petroleum-based polymers. Thus, the advancement of sophisticated bio-based materials is beneficial (Divya and Daniel 2021, Shorey and Mekonnen 2022, Singh et al. 2022, Mangal et al. 2023).

Gum rosin, a cost-effective renewable resource, is obtained through the distillation of resin excreted by pines and conifers. The principal components of gum rosin are resin acids, which include several types of acids such as dehydroabietic acid (DA), palustric acid, abietic acid, and neoabietic acid (Natsir et al. 2021). Gum rosin is often favored among bio-based and renewable raw materials due to its distinctive characteristics, including excellent adhesion and superior mechanical capabilities. Rosin can be employed as a plasticizer in several industries, including inks, adhesives, coatings, insulating materials, cosmetics, varnishes, medicines, and chewing gums, attributed to the reactive characteristics of carboxyl groups and/or conjugated double bonds

in hydrophenanthrene structures (De La Rosa-Ramírez et al. 2020, Parihar and Gaur 2022, De La Rosa-Ramírez et al. 2023). Nonetheless, the synthesis of rosin-based polymers exhibiting superior performance remains an enormous challenge. To broaden the applications of rosin in polymers, vinyl monomers, acrylic ester monomers, and allyl ester monomers generated from rosin have been synthesized (Mirabedini et al. 2020, Vevere et al. 2020, Yu et al. 2021, Jaswal et al. 2022).

Thermoplastic acrylic resins are high molecular weight polymers that soften and can be reshaped with the application of heat. They are generally used in road marking paints, surface coatings and spray paints. Single-component acrylic resins are widely used in industrial applications thanks to their ease of application, fast curing and fast drying properties. Conventional thermoplastic formulations typically include a thermoplastic resin and a plasticizer or toughening agent as components of their binder systems. Plasticizers frequently migrate to the surface of the paint, where they are then leached away, which has a detrimental effect on the performance of the paint throughout its service life (Felton and McGinity 1997, Vieira et al. 2011, Yerro et al. 2016, Jia et al. 2018, Taheri et al. 2019, Shukurov et al. 2021, Caillol 2023).

In this study, instead of introducing the plasticizer into the paint formulation, a bio-based gum rosin acrylic monomer as showing the plasticizer effect was utilized as an acrylic monomer to get acrylic copolymers that can be used directly without any plasticizer in the paint formulations. The paint coatings that are obtained from bio-based gum rosin acrylic copolymers, in which the gum rosin units are covalently bound and have a plasticizer effect, have been demonstrated to be glossier and look more lively, to have greater adherence, and to have a higher impact value.

2. Material and Methods

2.1. Experimental

Butyl acrylate (BAc) (Arkem Chemicals), styrene (Arkem Chemicals), acrylic acid (AA) (Sigma Aldrich, 99%), tolu-

ene (ISOMER, China), tert-butyl peroxybenzoate (TBPB) (AKPA),

toluene, methyl ethyl ketone and dichloromethane (Merck), tetrabutylammonium bromide, TBAB (Sigma Aldrich), Hydroquinone (Sigma Aldrich), GMA (Qingdao CBC Co. Ltd.), Gum rosin (Pınar Kimya) were used without any further purifications.

The chemical structure of the resin was confirmed using Attenuated Total Reflectance Fourier transform infrared spectroscopy (ATR-FTIR), with spectra obtained in the region $4000\text{--}700\text{ cm}^{-1}$ using a JASCO FT/IR-4200 spectrometer.

TA Instruments DSC250 dynamic differential scanning calorimetry, and a The Refrigerated Cooling System (RCS90) cooling controller were used for thermogravimetric analysis.

2.2. Gum Rosin Acrylate Synthesis

Gum rosin was used as a bio-based source as monomer in the synthesis of thermoplastic acrylates. In this study, gum rosin was first acrylated for this purpose to synthesize gum

rosin acrylate (GRA). The acrylation procedure and the reaction are given in Figure 1 and 2.

For GRA synthesis, 18.79 g glycidyl methacrylate (GMA), 40 g gum rosin (GR), 0.058 g Tetrabutylammonium bromide (TBAB) and 0.058 g hydroquinone mixture is dissolved in 60 ml Methyl Ethyl Ketone (MEK) solvent. The dissolved monomer mixture is taken into a 250 mL reaction flask. The temperature has increased to 60°C . The reaction time is 24 hours. At the end of the reaction, the MEK in the product is evaporated in a rotary evaporator. Extraction is done with dichloromethane (DCM): water in a 1:1 ratio. After purification, DCM is evaporated by using a rotary evaporator. The pure GRA product is obtained.

2.3. Thermoplastic Acrylate Synthesis

Thermoplastic acrylic copolymers were synthesized employing bio-based acrylic monomer at varying ratios (2.5%, 5%, and 10%), along with other commonly used monomers, styrene (50 g, 0.48 mol), butyl acrylate (40 g, 0.31 mol), and acrylic acid (1.2 g, 0.016 mol) in toluene solvent. Tert-bu-

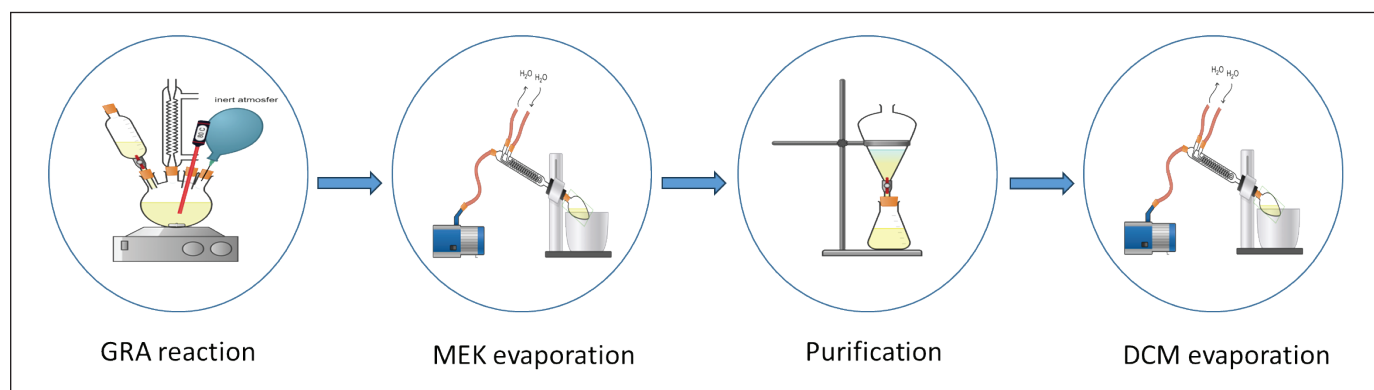


Figure 1. GRA synthesis scheme.

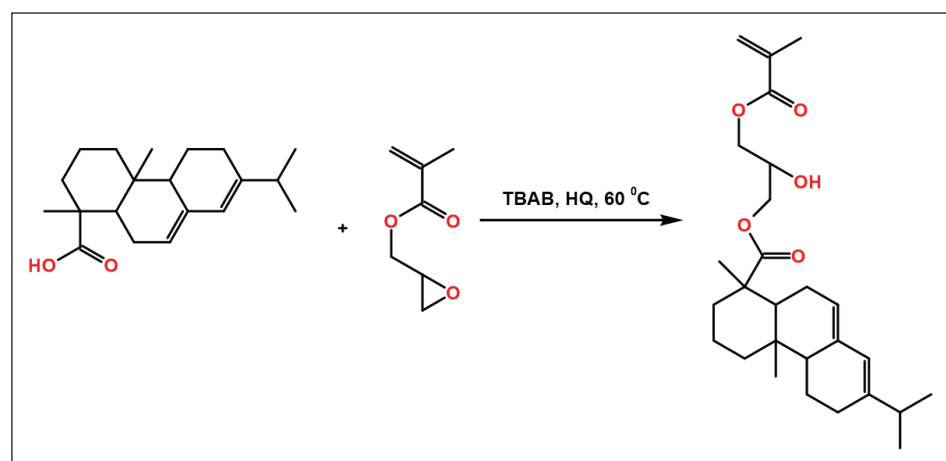


Figure 2. Synthesis of Gum Rosin Acrylate.

tyl peroxybenzoate (2.1 g, 0.010) was used as an initiator as 2% ratio. The solvent was taken into the reaction flask and heated to 110 degrees in a nitrogen atmosphere. The monomer mixture was dosed and the reaction was monitored by controlling the solid value. Solid analysis was performed according to the TS EN ISO 3251 test method. After the acrylate reached 60% solid value, the reaction was cooled. For the final product obtained, acid (mg KOH/g) was analyzed using the TS 2366 EN ISO 2114 method, viscosity (cP) was analyzed using the TS 6126 EN ISO 2555 method, and color (Hazen) was analyzed using the TS EN ISO 4630 method.

2.4. Thermoplastic Acrylate Surface Applications and Tests

In varnish and paint applications, gloss (ASTM D2457) and touch drying analyses were performed on paper plates; hardness (ASTM D4355) on glass surfaces; impact resistance (ASTM D2794) and adhesion tests (ASTM D3359) were performed on aluminum (Al), galvanized (Gal) and sheet metal surfaces. TQC Sheen BV., located in Capelleaan den IJssel, the Netherlands, provided the cross-cut adhesion test kit CC2000, which was utilized in order to evaluate the adhesion of dry coatings on their respective substrates. A Novo-Gloss Trio glossmeter was utilized in order to ascertain the level of brightness exhibited by the films. For determining the hardness, a TQC Sheen Pendulum Hardness Tester /SP0500 I141 equipment was utilized. The impact test with TQC Sheen was used in the processes of the mechanical tests. The formulation in Table 1 was used to prepare the dye mixture. A high-speed mixer was used to mix all of the paint's ingredients together. Glass and metal surfaces were painted using a film applicator that had a thickness of 90 micrometers.

Table 1. Paint formulation used for thermoplastic acrylate.

Material	%
Resin	40
Wetting and dispersing additive	0.5
Rheology additive	0.5
Titan	10
Calcite	15
Barite	23
Xylene	5.5
Butyl Acetate	5.5

3. Results and Discussion

3.1. Characterization

The characterization of the materials has been carried out by ATR-FTIR and DSC measurements. In Figure 3 (a), the synthesis of GRA was verified with FTIR spectra. The peak seen at 1637 cm^{-1} represents the C=C double bond in the acrylic group. The presence of tension in this region indicates that gum rosin is acrylated (Do et al. 2009).

Using DSC, the thermoplastic acrylate's glass transition temperatures (T_g) were ascertained. The T_g value, which is crucial for learning about the characteristics of the polymers, including their ability to be flexible or hard, was measured. Figure 3(b) displays the image.

The T_g of the blank thermoplastic acrylate was measured as $30.18\text{ }^{\circ}\text{C}$. As the gum rosin content of the samples increased, their T_g gradually decreased as 28.78 , 27.28 , and $24.12\text{ }^{\circ}\text{C}$ for the 2.5, 5 and 10%, respectively. The low T_g values, which also resulted in a poor crosslinking density, were probably caused by more flexible monomers in the sidechain or backbone of polymers. A decrease in T_g increased the polymeric network's flexibility and improved their adherence to the substrates (Lastovickova et al. 2021, Zhang et al. 2021).

3.2. Thermoplastic Acrylate Surface Applications

The physical and mechanical properties (solid ratio, color, viscosity, acid value, gloss, hardness, and drying time) of the resins and thermoplastic acrylate were given in the Table 2. Cross-cut and impact resistance tests and pull-off test images performed on aluminum, galvanized and sheet metal surfaces are given in Figure 4.

Better drying periods were seen when acrylate was applied with gum rosin, as indicated by the results in Table 2. This might be because gum rosin contains double bonds, which increase the structure's cross-linking. The final coatings' physical qualities are improved and the films become stronger because of this rise in crosslink density. Additionally, because moisture lengthens the drying times of paints, the hydrophobic characteristic of the gum rosin chemical composition may aid in reducing drying times. The gum rosin-based paint has very high gloss values despite having quick drying times. Gum rosin's chemical structure could be the cause of this. This also increased the adhesion effect to the surfaces (Soucek et al. 2012, Wilbon et al. 2013, Dizman and Ozman 2020). The adhesion of all coatings were enhanced with the integration of gum rosin acrylate.

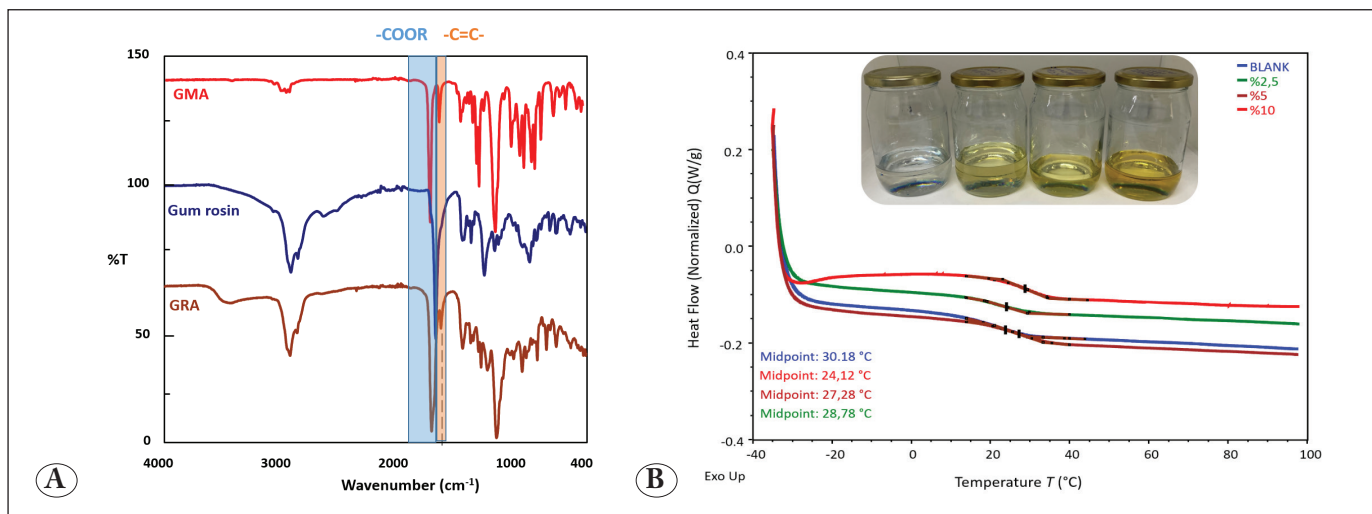


Figure 3. A) FTIR spectra of GRA and B) DSC curve of thermoplastic acrylate.

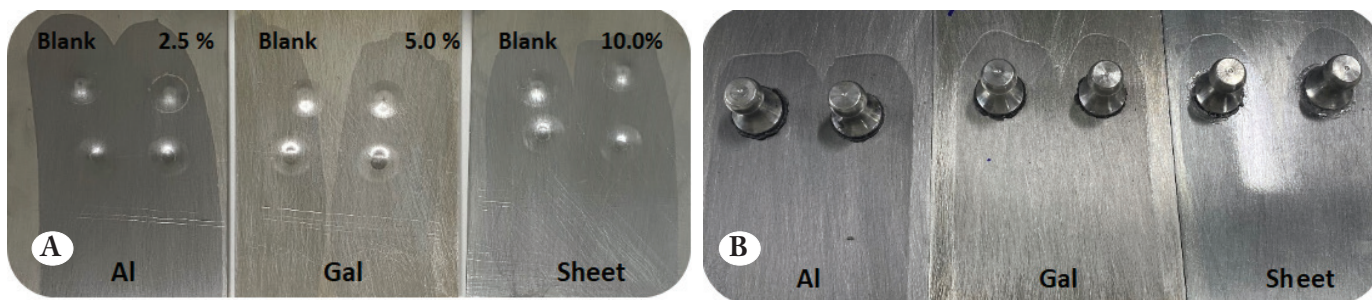


Figure 4. Images of (A) cross-cut and impact resistance and (B) pull-off tests.

Table 2. Mechanical and physical properties of the thermoplastic acrylate and paint.

Varnish				
Test	Blank	2.5%	5.0%	10.0%
Viscosity (Gardner/sec) ASTM D 1545-13	30	42	39	32
Acid Value (mg KOH/g) TS 2366 EN ISO 2114	7.97	9.67	8.27	7.59
Gloss ASTM D 2457	84	85	85	86
Persoz Hardness (1.day) ASTM D4355	147 P	102 P	96 P	73 P
Persoz Hardness (5.day) ASTM D4355	245 P	185 P	160 P	143 P
Drying Time (minutes)	15	10	10	10
Impact Strength (Al/Gal/Sheet) *ASTM D2794	2/3/2	1/1/2	1/1/2	1/1/2
Cross-cut Adhesion (Al/Gal/Sheet)*ASTM D3359	4/5/3	1/2/1	0/1/0	0/0/0
Pull-off Adhesion (MPa) ASTM D4541	1.04	1.72	1.86	1.92
Paint				
Gloss ASTM D 2457	24.5	28.8	36.5	44.8
Persoz Hardness (1.day) ASTM D4355	106 P	92 P	76 P	68 P
Persoz Hardness (5.day) ASTM D4355	208 P	195 P	163 P	132 P
Drying Time (minutes)	10	10	10	10
Impact Strength (Al/Gal/Sheet) * ASTM D2794	2/2/2	1/1/2	1/1/2	1/1/2
Cross-cut Adhesion (Al/Gal/Sheet) * ASTM D3359	4/3/5	3/2/3	1/1/1	0/0/0
*0 is the best, 5 is the worst				

Gum rosin acrylate contains hydroxyl groups that are capable of making more hydrogen bonds with the surface. Also throughout the incorporation of large gum rosin molecules in the polymer chains the interaction areas of the polymers also increases with the surface. That behavior let them contact with more connection points that enhances the adhesion. In the blank polymer, the pull-off test resulted 1.04 MPa whereas just only incorporation 10% gum rosin acrylate monomer in the polymer chains increased the adhesion strength to 1.92 MPa.

4. Conclusion and Suggestions

Consequently, a bio-based acrylic monomer was synthesized and utilized to get single component (1K) acrylic copolymers that contains different ratios (2.5, 5.0 and 10%) of GRA by using free radical polymerization. The varnish and paint formulations were prepared and the result showed that the incorporation of acrylic copolymers with increasing the amount of gum rosin acrylic monomer enhanced the coating properties positively in case of adhesion, glossiness, impact resistance and drying. The results indicate that increasing the quantity of gum rosin monomers in the polymers reduced the glass transition temperatures from 30.18 °C to 24.12 °C, which enhanced impact resistance and improved adhesion to metal surfaces. Adhesion is almost doubled while using 10% GRA monomer in compared to the blank. Impact resistance also increased. Surface hardness was reduced to its half due to the decrease in Tg values. Through this study, a bio-based acrylate monomer, which can be an alternative to petroleum-derived acrylic monomers, was synthesized and environmentally friendly polymeric materials suitable for sustainable and green chemistry were obtained.

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Author contribution: Cemil Dizman paper wrote, evaluated results, designed the analysis. Aleyna Turanlı experiment, collected data, paper wrote. Elif Cerrahoğlu Kaçakgil paper wrote, evaluated results, designed the analysis.

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