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Development of Hybrid Coatings for Anti-Corrosion Applications in Oil and Gas sector

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ABSTRACT
The physical, and mechanical properties as well as chemical and corrosion resistance of hybrid coatings comprising polyurethane/acrylic hybrids (PUA/AC) and acrylic polymers (AK) were investigated. Polyurethane (PUA) was synthesized through polyaddition polymerization of isocyanates [Isophorone diisocyanate (IPDI) and hexamethylene diisocyanate (HDI)] with polyols (GP 2000 and GP 4000) at an NCO/OH ratio of 0.85 and a temperature of 100°C. The acrylic copolymer (AC), based on methyl methacrylate (MMA) and butyl methacrylate (BuMA), was produced via bulk polymerization with benzoyl peroxide as a catalyst. The acrylic copolymer (AK) was prepared by grafting xanthan gum with styrene in various ratios (XG: St = 1:1.6, 1:5, and 1:8 w/w%). The results revealed that hybrid coatings demonstrated optimal chemical and corrosion resistance when PUA/AC hybrids were combined with AK. Enhancing both mechanical properties and corrosion resistance was achieved by integrating PUA/AC-10 with AK containing 8% styrene, resulting in superior anticorrosion performance including chemical and solvent resistances for the hybrid coating.

Keywords: polyurethane, acrylic, hybrid, coating, anti-corrosion

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Introduction

Metal corrosion in the oil and gas industry shortens equipment lifespan, causing financial losses and safety risks [[1], [2], [3], [4], [5]]. Polyurethane polymers (PUs), known for their renewable and versatile nature, are widely used in coatings to protect metals from corrosion, maintaining their critical role in this application [[6], [7], [8], [9], [10], [11], [12], [13], [13]]. Polyurethane polymers (PUs) are synthesized through the polymerization reaction between isocyanates and polyols [14].

Polyurethanes (PUs) have found immense success in the coating industry due to their enhanced mechanical and physical properties [10]. However, the emergence of new water-based polymers with lower volatile organic compounds (VOCs) has lessened the prominence of polyurethane resins in coatings, primarily for environmental reasons. Despite their eco-friendly nature, water-based coatings often suffer from high surfactant concentrations, which negatively impact surface characteristics [[1], [8], [15], [16], [17], [18]]. Additionally, both water-based and solvent-based polyurethanes exhibit low mechanical and physical performance. To address these limitations, modified polyurethane resins are developed by integrating modifiers into the polyurethane fiber structure through a hybrid process. Combining acrylics with PUs is expected to deliver significant performance enhancements in the resulting materials [[19], [20], [21], [22], [23]].

Acrylics and polyurethanes (PUs) offer notable advantages, such as excellent mechanical properties and strong chemical resistance [24]. Styrene and (meth)acrylates are commonly utilized to create hybrid materials with PUs, aiming to merge the benefits of different polymers for enhanced functionality. Polyurethane-acrylic latex has emerged as a promising alternative to PU-based coatings. By combining the unique features of PUs and polyacrylates, the resulting copolymer can exhibit desirable traits like rapid drying, strong substrate adhesion, high gloss associated with PUs, oxidative drying capabilities, efficient film formation, and the chemical resilience characteristic of acrylic latexes. However, the incompatibility between hydrophobic PU polymer units and the aqueous dispersions of acrylic polymers poses a challenge, limiting their effectiveness as blends in certain applications.

PU-acrylate hybrids often lead to reduced gloss and haze formation in films. To address this, polyurethane and acrylates are chemically bonded to produce PU-acrylic copolymer latexes. Negim et al. (2024) [25], however, explored an alternative approach by hybrid PU with 2-hydroxy ethyl acrylate to develop polyurethane-acrylic hybrids featuring an NCO/OH ratio of 2.2. Their findings revealed that, compared to pure PU, these hybrids' physical and mechanical properties improved as the proportion of 2-hydroxy ethyl methacrylate increased. The integration of acrylic components into the hybrids significantly altered the structure of the pure polyurethane and enhanced its final characteristics. PUA-acrylic hybrids were formulated by hybrid PUA with varying amounts of acrylic polymer to examine the impact of acrylic content on the hybrid films' mechanical and physical properties, as well as their chemical and corrosion resistance [26]. As a result, the hybrids demonstrated enhanced tensile strength, adhesion, hardness, and contact angle compared to the pure PUA and acrylic monomers. However, increased acrylic polymer content in the hybrid further elevated tensile strength, adhesion, hardness, and contact angle while reducing elongation at break. This was primarily attributed to the polymeric network formed by the cross-linking between PUA and acrylic polymer. Interestingly, hybrids with a PUA/acrylic hybrid composition containing 10% acrylic polymer displayed optimal chemical and corrosion resistance, making them particularly effective as coating materials [26]. The hybrid coating demonstrates an environmentally friendly profile, exceptional durability, and robust resistance to solvents and chemicals, along with superior mechanical properties. Additionally, the coating can be produced effortlessly without the need for specialized equipment. The work was further extended to include the application of the obtained acrylic copolymer based on xanthan and styrene compositions (XG: St - 1:1.6, 1:5, and 1:8 w/w%) to modify physicomechanical and anti-corrosion properties of the hybrid coating.

Experiments

Materials

Polypropylene glycol variants—GP-2000 (Mw = 2000 g/mol, OH number = 56 mg KOH/g), GP-4000 (Mw = 4000 g/mol, OH number = 29.5 mg KOH/g), GP-3000 (Mw = 3000 g/mol, OH number = 37 mg KOH/g), and GP - 2100 (Mw = 3000 g/mol, OH

Table 1 – Fee composition of polyurethane polymer (PUA)

	Wt (g)		Wt (%)
Polyols, OH			
GP 2000	168.5		82.2
GP 4000	21.6		10.5
Total	190.1		92.7
Mole of OH (gm/ mole)		0.0896	
Isocyanate, NCO			
IPDI	8.5		4.1
HDI	6.4		3.1
Total	14.9		7.3
Mole of NCO (gm/mole)		0.0762	
NCO/OH		0.85	

number = 56 mg KOH/g)—were sourced from Korea PTG, Korea. These were dried at 80°C and 1–2 mm Hg for 2 hours before use. Dibutyltin dilaurate (DBTDL), isophorone diisocyanate (IPDI), and hexamethylene diisocyanate (HDI) were procured from Bayer AG, Germany. Methyl methacrylate (MMA), butyl methacrylate (BuMA), Styrene (St), potassium persulfate($K_2S_2O_8$), and biopolymer xanthan gum (XG) were purchased from Sigma-Aldrich Chemical Co. (USA). Various solvents were utilized, including xylene, methyl ethyl ketone (MEK, purity > 99.9%), hydrochloric acid (ACS reagent, 37%), sulfuric acid (ACS reagent, 37%), ethanol (ACS reagent, 20%), and sodium chloride (ACS reagent, 10%), all obtained from Sigma Aldrich, USA. MOCA, a curing agent from TPUCO, Taiwan, was employed alongside ESOL N100 plasticizer from VISTALINE, Russia, and BYK-054, a defoamer from BYK, USA. Fillers such as calcium carbonate and pigments like TiO_2-R-996 were acquired from Elementis, Malaysia, and utilized without further purification.

Polyurethane polymer (PUA) Synthesis

The polyurethane (PUA) based on isophorone diisocyanate (IPDI), hexamethylene diisocyanate (HDI), and polyols (GP 2000 and GP 4000) were added to the reactor, and the mixture was heated at 100°C for 3 hours until the theoretical NCO value was attained, as evaluated by the di-n-butylamine titration technique (ASTM D 2572-19, 2019) [27]. The preparation of PUA and the methods of analysis have been previously described in a previous investigation [26]. Table 1 displays the samples that were made. The resulting PUA was clear and liquid, with viscosities of 120 mPa-s and 464.4 mPa-s at 5 and 50 rpm, respectively.

Synthesis of poly (methyl methacrylate-co-butyl methacrylate) (AC)

The preparation of copolymer based on methyl methacrylate (MMA) and butyl methacrylate (BuMA) in feed (5/5) and the methods of analysis have been previously described [26]. The MMA-co BuMA was liquid and transparent in appearance with viscosities of 432 mPa-s and 600 mPa-s at 5 and 50 rpm, respectively.

Synthesis of poly (xanthan gum -g- styrene) (AK)

The preparation of grafted copolymer based on xanthan (XG) and different ratios of styrene (St) (1:1.6 AK1, 1: 5 AK2, and 1: 8 AK3 w/w, %) respectively and the methods of analysis have been previously described [28].

Preparation of polyurethane/acrylic hybrids (PUA/AC)

PUA/AC hybrid was prepared by mixing process 90 % PUA and 10% AC at temperatures 60 °C and 600 rpm and labeled as PUAC-10.

Preparation of polyurethane/acrylic hybrids (PUACK)

Polyurethane/acrylic hybrids (PUACK) were prepared by mixing PUA/AC-10 and AK process at temperatures 60 °C and 600 rpm. Further details about the PUA/AK1 hybrids are given in Table 2.

Table 2 – Fee composition of the PUACK hybrids

Samples	PUAC-10 Wt., (gm)	PolyXG-g-St (AK) Wt., (gm)
PUACK1	90	10 (AK1)
PUACK2	90	10 (AK2)
PUACK3	90	10 (AK3)

Preparation of PUACK hybrid films

PUACK films were prepared by casting the solution onto a flat surface, followed by a curing process that lasted five days at room temperature. The cured films were then stored in a desiccator at ambient conditions to ensure proper preservation before undergoing characterization and measurement

Preparation of the PUACC and PUACK coatings

The preparation of PUACC (based on PUAC-10) and PUACK coatings followed specific formulations, with the weight percentages of the components detailed in Table 3. Across all formulations, the solid content of PUA or PUA/AC accounted for 27% of the total composition. Xylene and polyols (GP-3000 and GP-2100) were first combined in a vial and mixed for 10 minutes at 500 rpm. The plasticizer ESOL N100 and the defoamer BYK-054 were then incorporated into the mixture, followed by five minutes of stirring. Next, calcium carbonate (filler) and TiO_2-R-996 (pigment) were added, and the blending process continued for 30 minutes at a higher speed of 1200 rpm. Finally, the catalyst DBTL was introduced during the application of the coating onto the metal surface.

Table 3 – Anti-corrosion polyurethane/acrylic hybrid coatings (PUACC) and (PUACKC) formulations

Raw materials	Weight percent
PUAC-10 or PUACK	27
Xylene	6.45
GP-3000	9.24
GP-2100	4.5
ESOL N100	4.5
BYK-054	0.35
Calcium carbonate	40.16
TiO_2-R-996	4
DBTDL	0.4
Total	100

Application of the PUACC and PUACKC as coatings

Before coating application, metal samples measuring 9.0 cm × 0.9 cm × 15 cm were abrasively blasted and thoroughly cleaned. The PUACC and PUACKC-based coatings were applied using a film applicator, ensuring a wet film thickness of 75 µm. The coated samples were then left to cure at room temperature for 6 days.

Tests

The viscosity (η) of PUA/AC-10 and PUACK hybrids was determined using a Brookfield viscometer (Spindle 2) at rotational speeds of 5 and 50 rpm, maintained at a temperature of 25°C. The thixotropy index was calculated based on Equation 1.

$$\text{Thixotropy index (TI)} = \eta 5/\eta 50 \qquad (1)$$

The contact angle between water droplets and the sample surface was measured using a CAHN DCA-322 contact angle measuring device. The measurement was conducted at 25°C with a water droplet applied at a velocity of 100 lm/s. A small syringe was utilized to deposit the water droplet onto the surface being analyzed, and the contact angle was determined by observing the droplet formation on the monitor. The findings were derived by averaging three measurements conducted on distinct portions of the film. The tensile properties of the cast films were assessed using an MTS 10/M tensile testing system, operating at a crosshead speed of 50 mm/min with a 1-kN load cell. At least four values were averaged for accuracy. Additionally, the hardness of the films was determined using an indentation Barcol hardness tester by ASTM B648-10 [29]. Adhesion between the metal and hybrid polymers was evaluated using pull-off testing, as outlined in ASTM D4541-22 [30]. Corrosion resistance tests were performed on coated panels under various conditions, including exposure to salt (10% NaCl), base (10% NaOH), acid (37% HCl and H_2SO_4), and solvents (xylene, MEK, and ethanol), following ASTM D5402-93 [31] standards. Water resistance was assessed in compliance with ASTM D1647-89 [32]. Dry times were recorded at a stable temperature of 25°C.

Results and discussion

To enhance the mechanical properties of polyurethane/acrylic hybrid (PUA/AC-10) and improve their anticorrosion coating capabilities, copolymers derived from xanthan gum and styrene were prepared through grafting polymerization.

Viscosity and thixotropic index (TI) of PUA/AC-10 and PUACK

Figure 1 illustrates the effect of acrylic copolymer (AK), derived from xanthan gum (XG) and styrene (St), on the viscosity of polyurethane/acrylic hybrids (PUA/AC-10). The viscosity of PUA/AC-10 increases when combined with copolymer AK. As the content of styrene in AK increases, the viscosity of PUA/AC-10 increases from 476 mPa·s to 1105 mPa·s at 5 rpm and increases from 135 mPa·s to 290 mPa·s at 50 rpm which is attributed to the behavior of XG as a fluid [33]. Factors such as particle deformation, orientation of non-spherical particles, polymer chain alignment in the flow direction, and chain deformation contribute to this increase in viscosity [34]. Consequently, polyurethane/acrylic hybrids (PUACK) incorporating (XG-g-St) exhibit higher viscosity than PU/AC-10, due to the branched, cross-linked, and three-dimensional network structure of XG-g-St [35]. PUACK3, with 8% styrene, demonstrates the highest viscosities at 5 and 50 rpm, exceeding those of other polyurethane-acrylic hybrids [26]. The thixotropic index (TI) of polymer solutions is influenced by several factors, such as the type of copolymer, composition ratios, and polymer concentration [36]. Figure 2 shows the TI of PUACK across different ratios of St content. The results indicate that PUACK3, containing 8% of St, exhibited the highest TI value of 3.8, whereas PUACK2, with 5% of St, had the lowest TI value of 3.5. Thixotropy plays a significant role in shaping the formulations and preparation processes of coatings, which in turn affects their rheological properties [36]. These results are lower compared to those of polyurethane hybrids with acrylic polymers formulated using a 5:5 feed ratio of methyl and butyl methacrylate [26].

Adhesion

The adhesion of polyurethane to metal results from adsorbate layers and chemical bonds formed between isocyanate groups and the metal surface. Several factors influence polyurethane adhesion, including isocyanate content, polyol composition, and acrylic polymer properties [[37], [38]]. Figure 3 demonstrates the effect of AK on the adhesion of the PUA/AC-10 hybrid to metal. Results show that incorporating AK into the PUA/AC-10 hybrid enhances adhesion. While the adhesion strength of PUA/AC-10 is 6.9 MPa, the PUACK hybrid, formed by combining AK with PUA/AC-10, exhibits increased adhesion from 6.9 to 10.7.

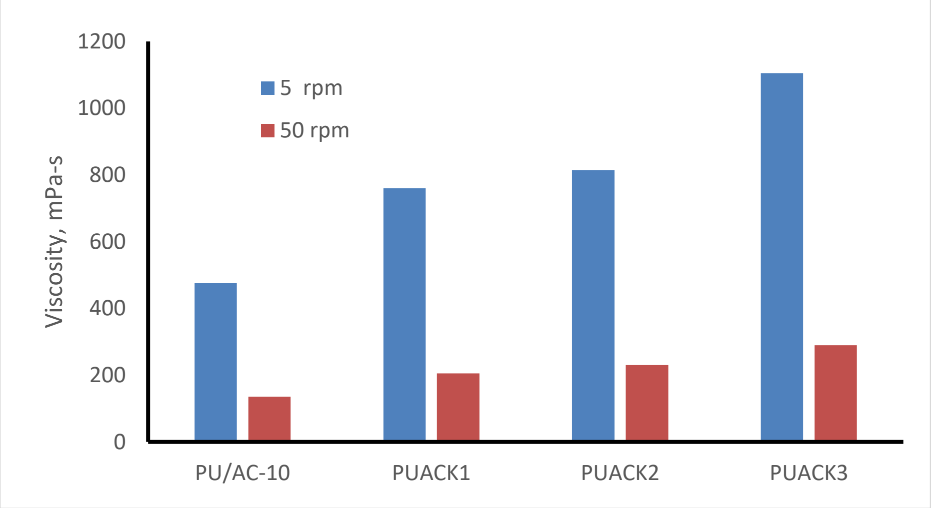

Figure 1 – The viscosity of PUA/AC-10 and PUACK hybrids

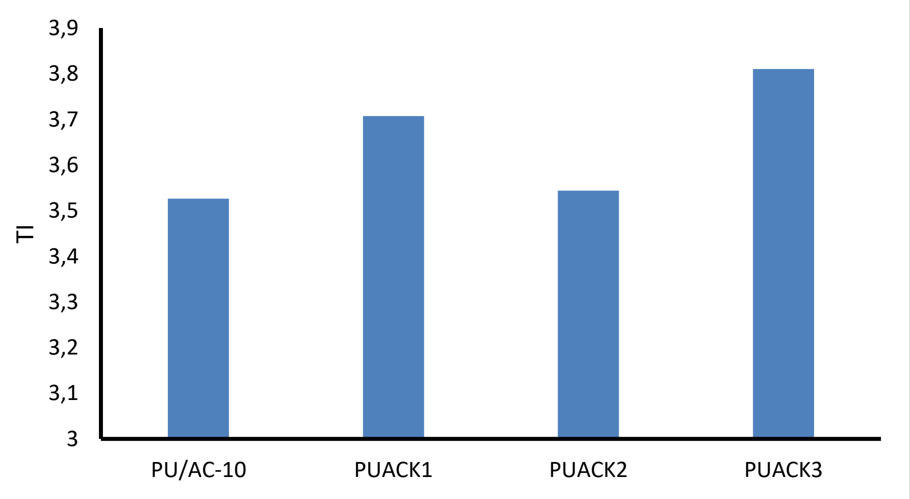

Figure 2 – TI of PUA/AC-10 and PUACK hybrids

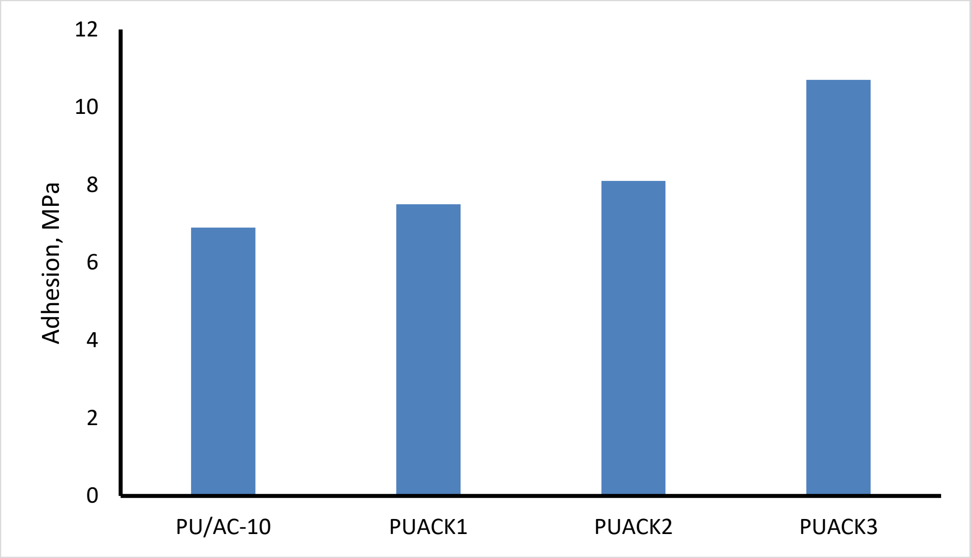

Figure 3 – Adhesion of PUACK hybrids on the metal

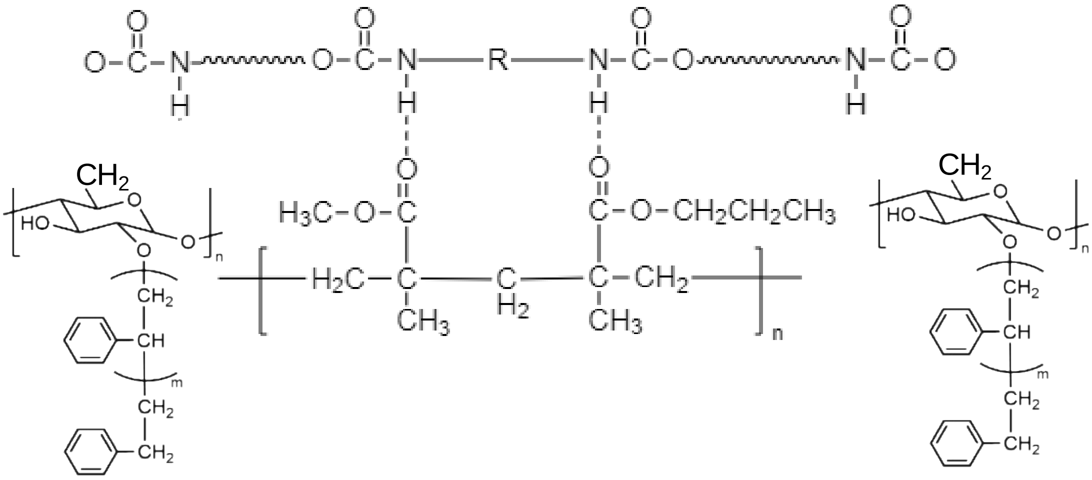

Figure 4 – The cross-linking between PUA, AC and AK in the PUACK hybrids

However, increasing the styrene (St) concentration in AK from 1.6% to 8.0% boosts adhesion strength from 7.5 MPa to 10.7 MPa. This improvement is attributed to crosslinking between PUA, AC, and AK, as depicted in Figure 4, which highlights the crosslinking between PUA and AK and the functional (NCO) groups on the substrate responsible for enhanced adhesion [[39], [40], [41]]. These results are lower compared to those of polyurethane hybrids with acrylic polymers formulated using a 5:5 feed ratio of methyl and butyl methacrylate [26].

Mechanical Properties

Table 4 shows the effect of acrylic copolymer AK with varying St compositions on the mechanical properties of the PUA/AC-10 hybrid. The results reveal that mixing AK with PUA/AC-10 significantly enhances the mechanical properties of PUACK, driven by crosslinking among PUA, AC, and AK and influenced by the side chain lengths forming hydrogen bonds between PUA, AC, and AK, as depicted in Figure 4. Increasing the St concentration in AK further boosts the mechanical properties of PUACK hybrids. For instance, the tensile strength of PU/AC-10 was 164 MPa, whereas PUACK improved tensile strength to 187.5 MPa for PUACK1 (St, 1.6%) and 245.1 MPa for PUACK3 (St, 8.0%). Additionally, hardness (shore D) increased from 53 for PU/AC-10 to 83.6 for PUACK3. All samples passed the impact test and crosshatch test, with PUACK displaying superior mechanical properties compared to PUA/AC-10 due to its dual crosslinking mechanism. Among PUACK hybrids, PUACK3 exhibited the best mechanical properties. Studies have consistently demonstrated that crosslinking the polymer backbone is an effective strategy for improving the mechanical properties of polymer films [42], [43], [44], [45]].

Table 4 – Mechanical properties of the PUA/AC-10 and PUA/AK hybrids

	Tensile strength, MPa	Elongation, %	Hardness, shore D	Contact angle	Impact test	Cross Hatch
PU/AC-10	164	125	53	132	Pass	Pass
PUACK1	187.5	105	65	135	Pass	Pass
PUACK2	195.9	91.8	71	149	Pass	Pass
PUACK3	245.1	88.2	83.6	151	Pass	Pass

Table 5 – The effectiveness of the hybrid coating against corrosion and chemical resistances reduced when the amount of St in AK in the hybrids increased.

Contact angle	PUACC-10	PUACKC1	PUACKC2	PUACKC3
Corrosion resistance				
NaCl (10%)	O	O	O	O
NaOH (10%)	O	O	O	O
HCl (37%)	O	O	O	O
H_2SO_4 (37%)	Δ	Δ	O	O
Water	O	O	O	O
Chemical resistance				
MEK	Δ	Δ	O	O
Xylene	O	O	O	O
Ethanol (20%)	O	O	O	O
Ethylene glycol	O	O	O	O
Wine	Δ	Δ	O	O
Acetone	Δ	Δ	Δ	O
Butyl alcohol	Δ	Δ	Δ	O

O: Suitable
Δ: Slight Suitable

Chemical and corrosion resistance

Table 5 demonstrates the chemical and corrosion resistance of hybrid coatings formulated with PU/AC-10 and AK to create anti-corrosion coatings (PUACKC). The chemical resistance of all coating samples was evaluated using MEK, xylene, ethanol (20%), ethylene glycol, wine, acetone, and butyl alcohol. Additionally, their corrosion resistance was assessed against NaCl (10%), NaOH (10%), HCl (37%), H_2SO_4 (37%), and water. The results indicate that hybrid coatings (PUACKC) based on AK exhibit superior chemical and corrosion resistance compared to hybrid coatings (PUACC-10) based on AC. Notably, PUACKC3, derived from AK3 with 8.0% St content, displayed the best anti-corrosion properties among the tested samples, attributed to the influence of the styrene ring on the mechanical properties of the polyurethane hybrid (PUACK).

Conclusions

Anti-corrosion coating hybrids were developed by combining PUA/AC-10 with AK to produce polyurethane/acrylic hybrids (PUACK) and examine how the AK content influenced the hybrid films' mechanical and physical properties, as well as their resistance to chemicals and corrosion. The inclusion of AK and PUA/AC-10 improved the mechanical and physical properties of the coating hybrids due to the functional groups present in PUA, AC, and AK, such as NCO, NH, carbonyl, styrene, and ester groups, which facilitate cross-linking among PUA, AC, and AK. The PUACK hybrids exhibited enhanced tensile strength, adhesion, hardness, and contact angle compared to PUA/AC-10. However, as the styrene (St) concentration increased, properties such as tensile strength, adhesion, contact angle, and hardness improved, while elongation at break decreased. This is attributed to the polymeric network formed through cross-linking among PUA, AC, and AK in the hybrid structure. Incorporating AK3 (St, 8.0%) into PUA/AC-10 further enhanced chemical and corrosion resistance compared to coatings based on PUA/AC-10.

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ТҮЙІНДЕМЕ

Гибридті жабындардың физикалық және механикалық қасиеттері зерттелді. Олардың құрамында полиуретан/акрил гибридтері (PUA/AC) және акрил полимерлері (АК) бар. Полиуретан (PUA) изоцианаттардың [изофорондицианат (IPDI) және гексаметилендиизоцианат (HDI)] полиолдармен (GP 2000 және GP 4000) 0,85 NCO/OH қатынасында және 100°C температурада поли біріктіретін полимерлеу арқылы синтезделді. Метилметакрилат (MMA) және бутилметакрилат (BuMA) негізіндегі акрил сополимері (AC) бензой пероксиді катализатор ретінде қолданылған көлемді полимерлеу әдісімен алынды. Акрил сополимері (АК) ксантан сағызы мен стиролды әртүрлі қатынаста (XG:St = 1:1,6, 1:5 және 1:8 мас.%) араластыру арқылы алынды. Нәтижелер көрсеткендей, PU/AC гибридтері мен АК үйлескен кезде гибридті жабындар оңтайлы химиялық және коррозияға төзімділік көрсетті. Механикалық қасиеттер мен коррозияға төзімділіктің жоғарылауына PU/AC-10-ды құрамында 8% стирол бар АК-мен біріктіру арқылы қол жеткізілді, бұл гибридті жабынның антикоррозиялық қасиеттерінің жоғары болуына әкелді.

Түйін сөздер: полиуретан, акрил, гибридті, коррозияға қарсы жабын.

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Разработка гибридных покрытий для антикоррозийного применения в нефтегазовом секторе

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АННОТАЦИЯ

Исследованы физические и механические свойства гибридных покрытий, содержащих полиуретановые/акриловые гибриды (PUA/AC) и акриловые полимеры (АК). Полиуретан (PUA) синтезирован путем полиприсоединительной полимеризации изоцианатов [изофорондиизоцианата (IPDI) и гексаметилендиизоцианата (HDI)] с полиолами (GP 2000 и GP 4000) при соотношении NCO/OH, равном 0,85, и температуре 100°C. Акриловый сополимер (AC) на основе метилметакрилата (MMA) и бутилметакрилата (BuMA) получен методом объемной полимеризации с использованием пероксида бензоила в качестве катализатора. Акриловый сополимер (АК) получен путем смешивания ксантановой камеди со стиролом в различных соотношениях (XG:St = 1:1,6, 1:5 и 1:8 мас.%). Результаты показали, что гибридные покрытия продемонстрировали оптимальную химическую и коррозионную стойкость при сочетании гибридов PU/AC с АК. Повышение механических свойств и коррозионной стойкости было достигнуто за счет сочетания PU/AC-10 с АК, содержащим 8% стирола, что привело к превосходным антикоррозийным свойствам гибридного покрытия.

Ключевые слова: полиуретановое, акриловое, гибридное, антикоррозийное покрытие.

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