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Hydrogel for Removal of Water Content from Liquid Fuels

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Abstract

Contamination of liquid fuels by water, which can be dissolved, suspended, or free, occurs due to spontaneous incorporation according to the composition of the fuel, the inherent use of water in the production processes, or by condensation and precipitation within transport and storage tanks. High-water content leads to microbiological development resulting in sludge formation, compromising the functioning of machines and engines; therefore, international regulatory agencies supervise the maximum water content. Currently, incomplete or energy-intensive processes are employed to remove water from fuels. Considering the need for a new technology that can remove water in all its state, while also being versatile for both industrial and domestic applications (in vehicle filters), hydrophilic polymeric hydrogels arise as a competitive technology for the dehydration of liquid fuels. Hydrogels used for removing water from biodiesel, diesel oil, marine diesel, and jet fuel in batch and continuous processes have been shown to meet international standard specifications with the advantage of being efficiently reused for several cycles. In this study, a review was made, which includes the main advances in the area.

Keywords: liquid fuels, hydrogels, water removal, diesel oil, biodiesel, marine diesel oil, jet fuel.

1. Introduction

Liquid fuels are the largest source of energy in the world, being used in transportation, commerce, industry, and domestic economies[1]. Due to its worldwide importance, specification standards are in place to regulate the quality of fuel available on the market. The

maximum water content is among the main parameters of fuel specification. Water contamination is one of the major concerns in production, handling, transporting, and storing fuels [2]. It occurs either by the direct contact of the fuel with water during reaction and separation steps of its production process or by contact with air of high relative humidity, in which the fuel spontaneously dissolves water. At room temperature, for example, fuels composed of hydrocarbons present a solubility of water ranging from 50 to 200 ppm; biofuels with structures containing hydrophilic polar groups, such as biodiesel, the solubility exceeds 1,300 ppm [3]. Moreover, the addition of biodiesel to diesel, for example, further increases the water retention capacity of the blend [4].

Considering the global efforts to reduce the CO₂ emission and even to increase performance of the final fuel, several countries have adopted public policies and legislative measures to increase the use of biofuels in their energy matrices. This significantly raises the concern in relation to water contamination; since the greater the polarity of the compounds in the mixture, the greater the hydrophilicity of the fuel [4]. The presence of water in fuels leads, most importantly, to microbiological activity and generation of sludge, which saturates filter elements faster and impairs the functioning of engine elements [5,6]. Technologies commercially available for water removal include gravitational equipment, centrifuges, and coalescing filters, which remove only free or suspended water. For the removal of dissolved water, energy-intensive processes are used such as heating and vacuum flash evaporators or packed bed of desiccant salts, which can lead to fuel contamination.

Considering the need for a new and complete technology that removes free and dissolved water and is simple to operate, at a competitive cost for industrial and domestic (integrated into vehicles) scales, hydrophilic polymeric hydrogels have been developed, showcasing their potential for removing water from liquids fuels, either in batches or in a continuous processes.

Generally, hydrogels can be used to dehydrated petroleum-based fuels, reaching lower water contents than that required by standard specifications. Specifically with diesel, good removal efficiency may be attained by employing even the most common type of hydrophilic hydrogels group, the neutral polyacrylamide. The use of ionic charged synthetic polymers as the poly(acrylamide-co-sodium acrylate) hydrogel were essential to achieve better water removal efficiencies from more hydrophilic fuels such as biodiesel and its blends.

2. Liquid Fuels

Crude oil and petroleum refining products—such as gasoline, diesel fuel, fuel oils, aviation kerosene, and marine fuels—as well as biofuels—such as ethanol and biodiesel—are

categorized as liquid fuels. Standard specifications regulate fuel quality, guaranteeing their performance and compliance with environmental restrictions, which has been increasingly demanded by the market. Among the main parameters of a fuel specification are viscosity, density, minimum flash point, maximum sulfur content, and maximum water content [7]. Water content may be a particularly challenging parameter to meet for some fuels since they can spontaneously dissolve water. Additionally, water is present everywhere in the fuel production and distribution chain; from being dissolved in the air to being used in industrial purification and cleaning processes. The solubility of water in fuel is a function of its chemical composition, temperature, and pressure to which it is subjected. Petroleum-derived fuels are hydrocarbons, consisting mainly of carbon and hydrogen; whereas biofuels are composed by hydrocarbon chains with oxygenated groups attached to them, such as acid, alcohol, and ester groups, a characteristic that gives biofuels a more hydrophilic behavior.

Currently, ethanol and biodiesel are the two most common types of biofuels in use. Although ethanol production is much higher, biodiesel production has grown faster since 2010, and more than quadrupled from 2010 to 2022 [8]. Biodiesel is a fuel consisting of fatty acid ester chains, generally produced via the transesterification reaction of vegetable oils and animal fats. Biofuels can offer considerable greenhouse gas emissions benefits, improve vehicle performance, and reduce demand for petroleum. For these reasons, several nations have implemented governmental policies and legislation to increase the use of biofuels in their energy matrices.

In the North American market—according to the US Energy Information Administration—the average percentage of biofuels added to gasoline, diesel, and jet fuel is expected to increase from 7.3%, in 2019, to 9.0%, in 2040 [9]. In the Brazilian market, the mandatory use of biodiesel in the mixture with diesel was set at 2% v/v, in 2005, reaching 12% v/v, in 2020 [10], with a predicted adjustment of up to 15% v/v, in 2023 [11]. These measures lead to an increase in the hydrophilicity of commercialized fuels, raising further concerns on the quality of fuels regarding water content.

2.1 Forms of water contamination in fuels

Water can be present in fuels in three states: dissolved, suspended, or free. Dissolved water is found when all the droplets are solubilized by the fuel and it is invisible to the naked eye, condition in which the fuel is referred as saturated. Suspended water appears as a dull, hazy, or cloudy appearance that takes time to coalesce or settle down [12]. When free water is present, there is a phase separation, and the water accumulates at the bottom of the tanks since it is denser than the fuel. Fuels are able to dissolve water spontaneously and have specific water

solubilization capabilities, especially considering polar group concentration, carbon chain length, temperature, and pressure. Generally, the greater the polarity of the compounds in the mixture, the greater the hydrophilicity of the fuel [4]. Table 1 presents the solubility of water in a few fuels at 20, 40, and 50°C.

Table 1 - Solubility of water in different fuels as a function of temperature.

Fuel	Solubility of water (ppm)			Reference
	20°C	40°C	50°C	
Biodiesel	1,720.0	1,873.0	1,980.0	[4]
Diesel oil	116.0	158.0	189.0	[4]
Marine diesel oil (MDO)	88.0	109.0	134.0	[13]
Jet Fuel – A	49.0	90.0	120.0	[12]

Biodiesel, for example, absorbs more moisture than petroleum diesel due to the hygroscopic nature of its fatty acid ester chains. The addition of biodiesel to fossil diesel increases the water holding capacity of the mixture. Pure biodiesel is capable of absorbing up to 1,980 ppm of water at 50°C, which is 10 to 15 times higher than pure diesel at the same temperature [4]. Although water can be considered a fuel contaminant, its use is inherent to many fuel production processes. During oil refining, steam is injected into the distillation columns for product rectification. In biodiesel production, wet washing is the most used technique to remove impurities, such as catalyst, glycerol, and residual alcohol, which are produced in the transesterification reaction. The water content of biodiesel that is purified through a wet washing method is typically higher than 1,000 ppm, and further reduction of the water content is required to meet the standard specification [14].

Other means of water contamination of fuels include condensation of air humidity on the walls in storage tanks, precipitation of dissolved water from the fuel itself at low temperatures, water left behind after cleaning operations in tanks or transport vehicles, and rainwater that can enter through poorly sealed tanks [12]. In practice, the exposure of fuels to water is inevitable. Water contamination can occur during the production process, transportation, and in storage, either by direct contact of the fuel with water or by contact with air of high relative humidity.

2.2 Effect of water on fuel processes and engines

The presence of water in fuel oils can result in numerous operational problems from its production to its use in vehicles. The presence of water in the biodiesel production process

reduces the activity of the catalyst and can lead to the hydrolysis of vegetable oil and glycerol to free fatty acids, which react with the catalyst to produce a saponified product [15]. Soap formation and reduced catalyst effectiveness result in low biodiesel conversion, in addition to hampering the product separation and purification stages [16].

The consequences of this contaminant in fuel supply systems include the foam formation in tanks, cavitation at pump suction, and corrosive action [17]. In the final product, growth of microorganism are suggested, which generate films and lead to clogging of filters, reducing the stability and the specific heat of the product [18]. These effects compromise the functioning of motor vehicle components, resulting in loss of propulsion and corrosion of equipment. Regulatory agencies, such as the American Society for Testing and Materials (ASTM) and the Brazilian National Agency for Petroleum, Natural Gas, and Biofuels (ANP), establish fuel specifications to ensure product quality. Table 2 shows the maximum water content established by these agencies for different fuels. Notably, these regulatory agencies adopt test methods that differ in their accuracy of detecting the water content in fuels. For biodiesel, for example, the methods allowed by ASTM D6751 are the ASTM D2709 or D1796, which are tests based on centrifuging the sample and reading the meniscus of a conical test tube. The ANP Resolution No. 45 (08/25/2014) allows the analysis method ASTM D6304, a technique that employs automatic volumetric Karl Fischer titration to detect traces of water.

Table 2 - Maximum water content for some fuels according to ASTM and ANP Resolution.

Fuel	International specification	Limits¹ (ppm)	ANP Resolution	Limits¹ (ppm)
Biodiesel	ASTM D6751	500.0	No. 45 (2014/08/25)	250.0 (manufacturer) 350.0 (distributor)
Diesel fuel	ASTM D95	500.0	No. 50 (2013/12/23)	200.0
Marine diesel oil (MDO)	ISO 3733	3,000.0	No. 52 (2010/12/29)	3,000.0

¹ Limits converted to ppm.

2.3 Technologies for removing water from fuels

Depending on the fluid characteristics—such as droplet size; water concentration; operating conditions, such as the water removal requirement; and process feed flow rate—different water separation technologies are commercially available and have been industrially employed for removing water from fuels.

Gravitational separators are based on the different densities of fluids, being applied to remove only free water. The size of conventional separators is indicated by the retention time required for gravitational separation to occur; this time is directly proportional to the volume of fluid. Thus, gravitational separators have low efficiency since high residence times, and therefore, large vessels, are required for the small droplets of free water to coalesce into drops large enough to migrate to the oil-water interface [19].

Centrifugal separators are used to remove free and lightly suspended water and provide efficient separation with short residence times for batches and continuous processes [20]. The limitations of centrifuges include high operating and capital costs. They are energy-intensive process and do not remove dissolved water from the fuel. Coalescing separators, or simply coalescers, assist small water droplets to combine into larger droplets, separating from the oil more easily. Despite having operational advantages, when compared with centrifuges, such as lower energy consumption, more compact units, and high efficiency in separating low-viscosity fluids, they are also unable to remove dissolved water, and they are sensitive to fluids that have solid impurities, which can clog the coalescing membranes and reduce its efficiency [20].

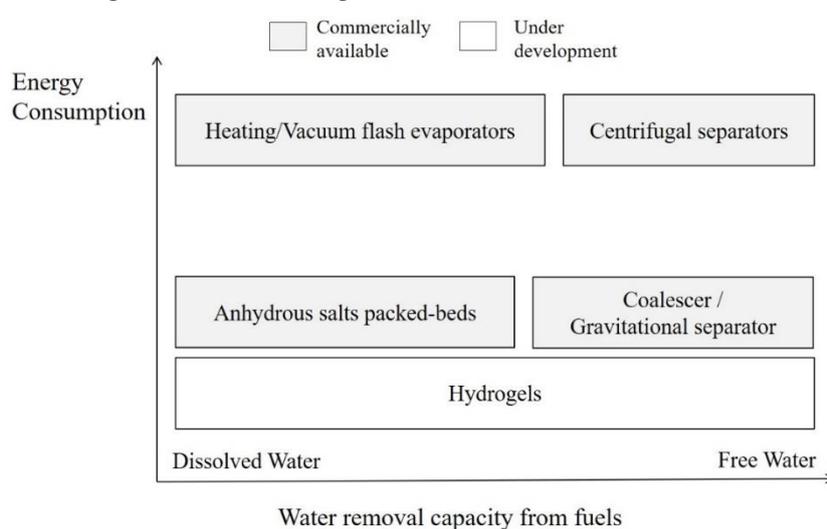
Anhydrous salts packed beds are units containing solid dehydrating compounds capable of removing free and dissolved water. The salts combine with the water in the fluid to form an aqueous solution which can be separated from the fuel flowing through the unit. Sodium chloride is the cheapest option employed in industrial process but it reduces the residual water level to about 75% of relative saturation; whereas calcium chloride, which is normally considered a highly cost-effective combination, is capable of removing about 50% of the saturation level [21]. Although drying salts are efficient for removing water from fuels, they require replacement of the salt beds used in the process, introducing the needs for maintenance interventions. Additionally, the generation of brine is inherent to this process, leading it to be sent to effluent treatment plants. Furthermore, the contamination of fuels with sodium, potentially originated from drying salts, was proposed as a relevant factor in incidents of filter clogging and fouling in nozzles [22,23].

Vacuum flash evaporators can also effectively remove free, suspended, and dissolved water from fuels. The technique consists of heating the oil to approximately 60°C and spraying it through a nozzle, into a vacuum chamber of 0.6–0.7 bar [24]. The combination of temperature and vacuum causes water to flash to steam at a temperature low enough to not cause thermal damage to the oil. Fuel dispersion makes the drying process more efficient; dry fuel droplets are collected at the bottom of the vacuum chamber. The main disadvantage of these dryers is

the need for a vacuum system that can pump steam out of the chamber, meaning expensive investments in equipment and high energy operating costs [25].

Thus, commercially available technologies employed to remove free water from fuels include centrifugal separators, gravitational separators, and coalescing filters (coalescers). For dissolved water heating/vacuum flash evaporators and anhydrous salt packed beds are frequently employed. However, each of these technologies have limitations, such as high energy and installation costs, possibility of fuel contamination, or low dissolved water removal efficiency [26]. Thus, hydrophilic polymeric hydrogels emerge as a promising technology for removing water from fuels; they are able to remove both free and dissolved water, are easy to operate, have a competitive cost, and can be efficiently reused and applied in industrial and domestic scale. Figure 1 shows a comparison among the commercially available technologies and hydrogels based on their energy consumption and water removal capacity.

Figure 1 - Technologies for water removal from fuels.



Source: Authors, 2022

Alt-Text: Commercially available technologies to remove free water include centrifugal separators—which are an energy-intensive technique—; gravitational separators; and coalescers. For dissolved water, vacuum flash evaporators are used, which is a process of high-energy demand, or anhydrous salts packed-beds are employed. Hydrophilic hydrogels are a promising technology that remove both free and dissolved water and have lower energy consumption when compared with commercial technologies.

3. Application of hydrogels for water removal from liquid fuels

On account of their hydrophilic characteristics, polymeric hydrogels have been applied to remove water from liquid fuels [13,26–37]. They are cross-linked three-dimensional materials capable of retaining a large amount of water in their structure [32]. Hydrophilic functional

groups that are bonded to the main chain of the polymer confer water-retaining abilities to the hydrogels; while its elastic properties and mechanical resistance, as well as the insolubility of the polymer matrix in an aqueous medium, result from the magnitude of the cross-linking of the network [38]. Among the advantages of applying systems with hydrogels to remove water from fuels, it is possible to highlight their versatility regarding the polymeric composition, which allows obtaining different polymeric matrices, according to the need for the external stimulus to which the hydrogel will be subjected; thus obtaining free and dissolved water removal [26,32] and good chemical stability. Additionally, the hydrogels can be recovered, regenerated, and reused [39,40].

Synthetic polymers systems have been applied to remove water from fuels due to their usual non-toxic and inert nature, long chain lengths, ability to preserve their shape and mechanical strength, and convenient adjustment of their mechanical, chemical, and biophysical properties [41]. Those materials are usually obtained by free radical polymerization—which is the most commonly employed cross-linking method for hydrogel synthesis—with the advantages of high reactivity, high conversion, and reaction under moderate conditions [42]. Synthetic homopolymer hydrogels as polyacrylamide, sodium polyacrylate, poly(acrylic acid), and copolymers hydrogels polyacrylamide-based—being poly(acrylamide-co-sodium acrylate) (poly(AAm-co-SA)), poly(acrylamide-co-acrylonitrile) (poly(AAm-co-AN)), and poly(acrylamide-co-acid acrylic) (poly(AAm-co-AA))—have been employed to remove water from liquid fuels, such as diesel, marine diesel, and biodiesel and its diesel blends [13,26–37,43,46,48]. Hybrid sources of hydrogel, which contain a natural and synthetic polymer using *Moringa oleifera* loaded keratin-co-sodium acrylate, has also been successfully applied for diesel purification [43]. Jet fuels present very low water affinity and, to remove traces of water, Li et. al (2021) have used poly(N-isopropyl acrylamide) (PNIPAm) hydrogel containing hygroscopic chloride polypyrrole (PPy-Cl); they were able to obtain high removal efficiencies even after several dehydration cycles. Thus, the results reported in the literature have demonstrated that polymeric hydrogels are promising materials for removing both free and dissolved water from liquid fuels, either in batches or in continuous systems, as well as for being reusable.

3.1 Hydrogels for removing water from petroleum-based fuels

Free, suspended, and dissolved water are satisfactorily removed from petroleum-based fuels employing hydrogels, reaching comfortable water levels—lower than those required by specifications. Due to their non-polar structure and lower water affinity in comparison to polar fuels, petroleum-based fuels, such as diesel, has been successfully purified even by applying

the most common type of hydrophilic hydrogels group, the neutral polyacrylamide [31], [32]. In this case, 1 to 2 h of contact with diesel were enough for the hydrogel to absorb dissolved water and reduce it to the levels allowed by standard specification (<100 ppm).

Traces of dissolved water in petroleum-based jet fuel could be reduced from 47 to 7 ppm within 6 h of using poly(N-isopropyl acrylamide) / chloride polypyrrole hydrogel [27]. Table 3 and Table 4 show the results and experimental conditions of studies employing hydrogels to remove water from petroleum-based fuels, either in batches or in continuous processes, respectively. In the batch processes, 80.4% and 98.2% efficiencies were achieved in the removal of free, suspended, and dissolved water from diesel oil (initial water content of 350 ppm) and marine diesel oil (initially at 5,000 ppm) respectively, by using polyacrylamide [32] and poly(AAm-co-SA) hydrogels [13], in that order. Diesel oil containing only dissolved water (initial concentration ranging from 170 to 240 ppm) obtained water content levels lower than 100 ppm when treated with neutral polyacrylamide hydrogels [31,32], poly(AAm-co-SA) and poly(AAm-co-AA) [26].

Studies focused on fuel water removal with hydrogels in continuous processes are particularly interesting since they address industrial operating conditions. Particles of poly(AAm-co-AA) and poly(AAm-co-SA) hydrogels filled in a random packing column showed similar performances, removing 50.0% water from saturated diesel oil, with a flow rate of 1 ml/min [34]. Furthermore, an unprecedented study demonstrated the ability of a packed bed filled with lyophilized sodium polyacrylate hydrogel continuously treat cloudy diesel (Figure 2). The fixed bed containing 4 g of hydrogel was able to reduce the turbidity of more than 5 L of diesel under a 6.5 ml/min flow rate, reducing the water content to about 100 ppm [35], meeting the regulatory requirement regarding the permissible water content for this fuel.

3.2 Hydrogels for removing water from biodiesel and its diesel blends

The use of hydrogels to remove water from more hydrophilic fuels such as biodiesel and their diesel blends have been similarly investigated in batches (Table 5) and in continuous processes (Table 6).

In batch process, water contents from 320 to 1,100 ppm were obtained in saturated biodiesel (initial contents from 1,300 to 2,500 ppm) by applying homopolymer hydrogels such as polyacrylamide, sodium polyacrylate, poly(acrylic acid), and copolymers, such as poly(AAm-co-SA), poly(AAm-co-AN), and poly(AAm-co-AA). The efficiencies of removing water from saturated biodiesel varied according to hydrogel composition, hydrogel mass/biodiesel volume ratio, and temperature employed in this process. In the special case of saturated biodiesel, the use of ionic charged synthetic polymers, such as anionic

polyacrylamides hydrogels, were essential to achieve better efficiency. Anionic polyacrylamides comprise the class of polymers that contain acrylamide monomer at some level, copolymerized with acrylate salts derived from acrylic acid [44], such as the poly(AAm-co-SA). This material has greater water removal capacity due to the presence of dissociated ionic charges in the hydrogel structure, which results in an increase in the osmotic driving force due to the strong ion-dipole interaction. Water content below than that specified by ASTM for biodiesel was reached by applying a moderate ratio of poly(AAm-co-SA) hydrogel mass/fuel volume, resulting in a reduction from 1514 to 453 ppm in a batch process [37]. Water content lower than the ASTM and ANP standards (for distributors) were also achieved by hydrogels in systems with temperatures around 40°C [28,45]. Owing to its hydrophilic behavior, additional efforts have been put into hydrogels design in combination with process parameters setup to meet standard specification for biodiesel, which will be discussed later in this study.

Regarding diesel-biodiesel blends, neutral polyacrylamide hydrogels reduced the water content of the saturated mixture containing 20% (v/v) and 40% (v/v) of biodiesel, B20 and B40 respectively, to levels below those allowed by ASTM [31]. However, under the same experimental conditions, the polyacrylamide hydrogel could not meet the specification requirements for diesel blends above 60% in biodiesel, due to its hydrophilic structure.

Regarding the removal of water from biodiesel in a continuous process, poly(AAm-co-SA) and poly(AAm-co-AA) hydrogels continuously removed free water in a bed named by the authors as Hydrogel Discs on Sieve Trays (HDST) [36] (Figure 3). Hydrogels in the form of perforated discs were placed on polyamide 12 sieve trays produced by additive manufacturing. Efficiencies higher than 97.0% were observed varying the flow rate from 1 to 3 ml/min, obtaining turbidity-free biodiesel in the output stream. The system has demonstrated high potential for use as a guard bed [36]. Dissolved water was also removed from biodiesel in a packed-bed containing poly(AAm-co-SA) and poly(AAm-co-AA) hydrogels, water content was reduced from 1,500 to around 750 ppm [34]. Notably, while US legislation accepts up to 500 ppm of water in biodiesel, measured through visual meniscus reading techniques, Brazilian legislation has much stricter criteria regarding the maximum admissible water content (250 ppm for producers and 350 ppm for distributors). A biodiesel quality control study conducted in Brazil observed water contents higher than 800 ppm along the biodiesel logistics: from the producer in the state of Mato Grosso (where water content was initially observed of 160 ppm) to the distributor located in the city of Manaus, in the state of Amazonas (distance of 2,514 km) [46]. The humid climate of some regions added to the hydrophilic character of biodiesel makes

the maintenance of this content below 350 ppm a current challenge to be faced by distributors of biodiesel in Brazil.

Table 3 - Application of hydrogels for removing water from petroleum-based fuels in batch processes.

Reference	Fuel	Water form	Hydrogel composition	Ratio hydrogel/fuel (mg/ml)	Contact time (h)	Initial water content (ppm)	Removal efficiency (%)
[31]	Diesel (B0)	Dissolved	Polyacrylamide	10.0	1	220	60.00%
[32]	Diesel	Dissolved	Polyacrylamide	50.0	2	240	65.00%
		Free and Dissolved				350	80.40%
[26]	Diesel	Dissolved	Poly(acrylamide-co-sodium acrylate)	50.0	24	170	59.20%
			Poly(acrylamide-co-acid acrylic)				58.30%
[43]	Diesel	Free and Dissolved	<i>Moringa oleifera</i> loaded keratin-co-sodium acrylate	80.0	3	18,000	95.00%
[27]	Jet Fuel	Dissolved	Poly(N-isopropyl acrylamide) / chloride polypyrrole	10.0	6	47	85.10%
[13]	Marine diesel oil	Free and Dissolved	Poly(acrylamide-co-sodium acrylate)	20.0	2	5,000	98.20%
			Poly(acrylamide-co-acrylonitrile)				98.00%

Table 4 - Application of hydrogels for removing water from petroleum-based fuels in continuous processes.

Reference	Fuel	Water form	Hydrogel composition	Flow rate (ml/min)	Packed-bed characteristics	Initial water content (ppm)	Removal efficiency (%)
[34]	Diesel	Dissolved	Poly(acrylamide-co-sodium acrylate)	1.0	D = 30 mm; L _{packed-bed} = 3 mm; Porosity = 0.4	N/D	~50%
		Dissolved	Poly(acrylamide-co-acid acrylic)				~50%
[35]	Diesel	Suspended and Dissolved	Sodium polyacrylate	3.0 - 6.5	Di = 17 mm L _{packed-bed} = 45-60 mm	630	84.0%

Table 5 - Application of hydrogels for removing water from polar fuels in batch processes.

Reference	Fuel	Water form	Hydrogel composition	Ratio hydrogel/fuel (mg/ml)	Contact time (h)	Initial water content (ppm)	Removal efficiency (%)
[30]	Biodiesel	Dissolved	Poly(N-isopropylacrylamide) (pNIPAM)	5.0, 10.0 e 12.0	24	1,800	47.0- 58.0%
			Poly(NIPAM-co-vinyl propionate)				38.0 - 58.0%
			Poly(NIPAM-co-butylacrylate)				23.0 - 27.0%
			Poly(NIPAM-co-vinyl biphenyl)				36.0-58.0%
			Poly(NIPAM-co-vinyl laurate)				36.0-58.0%
			Poly(NIPAM-co-vinylpyridine)				25.0-34.0%
			Poly(NIPAM-co-acrylic acid)				42.0-58.0%
			Poly(NIPAM-co-vinyl laurate-coacrylic acid)				38.0-55.0%

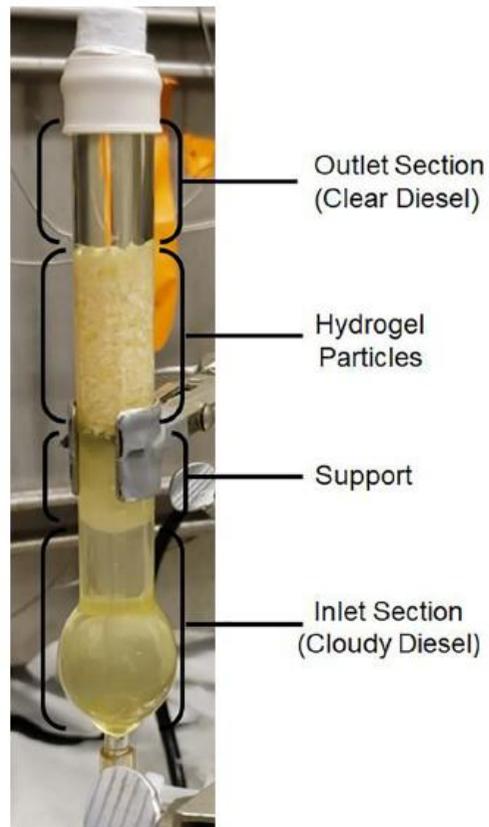
Reference	Fuel	Water form	Hydrogel composition	Ratio hydrogel/fuel (mg/ml)	Contact time (h)	Initial water content (ppm)	Removal efficiency (%)
[31]	Biodiesel	Dissolved	Polyacrylamide	10	4	2,200	63.60%
	Diesel-Biodiesel (B60)					~ 1,270	58.00%
	Diesel-Biodiesel (B40)					~ 750	57.00%
	Diesel-Biodiesel (B20)					~ 550.0	64.00%
[32]	Biodiesel	Suspended and Dissolved	Polyacrylamide	50	13	2,100	63.30%
[33]	Biodiesel	Dissolved	Poly(acrylamide-co-sodium acrylate)	125	18.3	2,100	39.50%
[28]	Biodiesel	Dissolved	Poly(acrylamide-co-acrylonitrile)	4	72	1,219.0–1,303.0	62.0 - 74.0%

Reference	Fuel	Water form	Hydrogel composition	Ratio hydrogel/fuel (mg/ml)	Contact time (h)	Initial water content (ppm)	Removal efficiency (%)
[26]	Biodiesel	Dissolved	Poly(acrylamide-co-sodium acrylate)	50	24	2,583	53.40%
			Poly(acrylamide-co-acid acrylic)				35.00%
[29]	Biodiesel	Suspended and Dissolved	Polyacrylamide	4	24	6,977	82.10%
			Sodium polyacrylate				84.30%
			Polyacrylic acid				40.80%
[37]	Biodiesel	Dissolved	Polyacrylamide	9.3- 500.0	48	1,514	29.0 – 42.0%
			Poly(acrylamide-co-sodium acrylate)				42.0 – 70.0 %
			Poly(acrylamide-co-acid acrylic)				33.0 – 52.0 %
[47]	Biodiesel	Dissolved	Poly(acrylamide-co-acrylonitrile)	4	72	1,333	62.0% – 74.0%

Table 6 - Application of hydrogels for removing water from polar fuels in continuous processes.

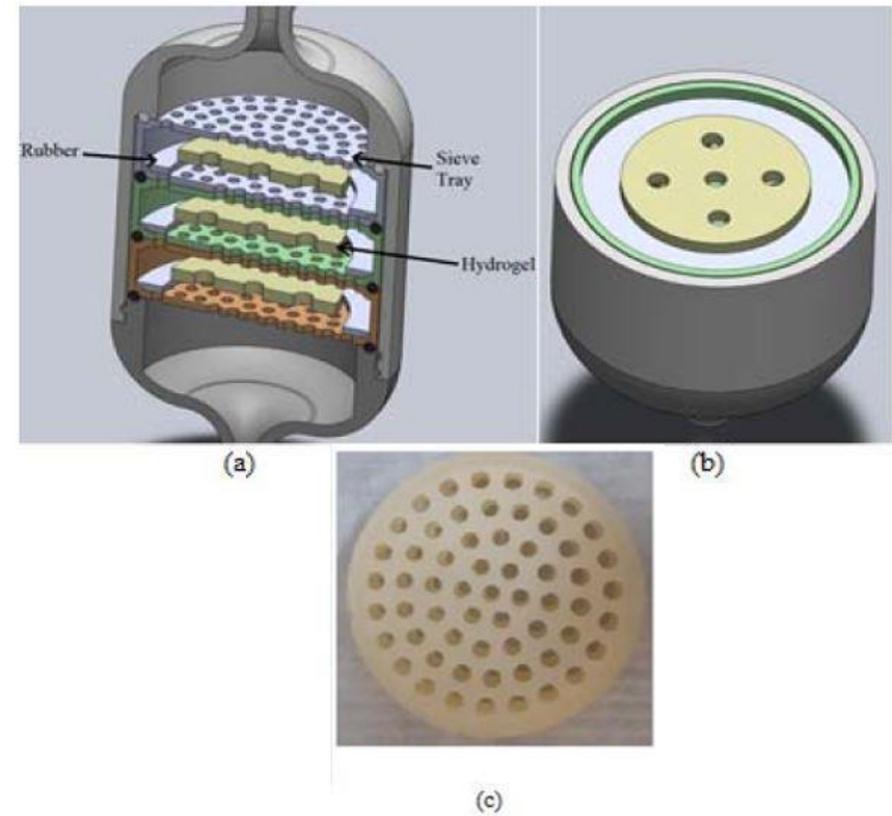
Reference	Fuel	Water form	Hydrogel composition	Flow rate (ml/min)	Packed-bed characteristics	Initial water content (ppm)	Removal efficiency (%)
[34]	Biodiesel	Dissolved	Poly(acrylamide-co-sodium acrylate)	1	D = 30 mm; L _{packed-bed} = 3 mm; Porosity = 0.4	1,500	~54.0%
		Dissolved	Poly(acrylamide-co-acid acrylic)				~54.0%
[36]	Biodiesel	Dissolved	Poly(acrylamide-co-sodium acrylate)	1 - 3	Hydrogels discs (D = 26 mm with 5 holes of 2.0 mm) placed on sieve trays of polyamide 12 of D = 40.0 m with holes diameter of 2.0 mm.	2,042	31.60%
		Free				50,000 and 100,000	98.00%
		Dissolved	Poly(acrylamide-co-acid acrylic)			2,042	26.00%
		Free				50,000 and 100,000	98.00%

Figure 2 - Fixed bed containing sodium polyacrylate hydrogel particles. Adapted with permission from [35], Copyright © (2022) Elsevier Ltd.



Text-Alt: Fixed bed containing sodium polyacrylate hydrogel particles treating a cloudy diesel inlet stream. After passing through the hydrogel bed, the diesel stream appears completely clear.

Figure 3 - Bed scheme of hydrogel discs supported on sieve trays. Internal view (a), top view (b) and sieve tray (c). Adapted with permission from [36], Copyright © (2021) AIDIC/CET Servizi S.r.l.



Text-Alt: (a) HDST contains compartments in the form of sieve plates, with holes. Each compartment contains a perforated hydrogel disc with 5 holes. (b) Top view of hydrogel disc contain 5 holes. (c) Sieve tray used as support for hydrogel discs.

3.3 Hydrogels design for water removal from liquid fuels

The main characteristics required of hydrogels to remove water from fuels in industrial processes are (1) high water removal capacities and (2) good mechanical strength in their swollen state. The first feature (1) is essential for higher water removal efficiency from hydrophilic fuels as biodiesel.

Thus, considering the fuels physical and chemical characteristics and their specific dewatering requirement in industrial processes, those hydrogels attributes have been designed by tuning their chemical composition and structural network parameters, as well as combining them with process conditions, such as hydrogel mass/volume fuel ratio and temperature.

3.3.1 Ionic charged hydrogels for high water removal capacities

Most published studies involving hydrogels for fuel dehydration applications are based on polyacrylamide uses in its pure form or copolymerized with other monomers. Polyacrylamide has stability characteristics, it is inert and presents viscoelastic and hydrophilic properties in addition to competitive price, which makes it commercially relevant due to its versatility. However, as we have previously emphasized, hydrogels with higher removal capacity have been shown to be indispensable to remove water from biodiesel and its blends with diesel due to their greater affinities with water.

When a polymeric network is immersed in a fluid, the network begins to absorb water due to the thermodynamic compatibility of the polymeric chains and water. Opposing this osmotic force, there is an elastic retractive force, induced by the cross-linking of the network [48]. At equilibrium, there is no additional water absorption due to the balance between elastic and osmotic forces. In the case of ionic hydrogels, ionization degree of the polymer chains and ionic strength of the external solution should be considered. Water absorption phenomenon can be described in terms of the change in the chemical potential of water at constant temperature and pressure as:

$$\mu_1 - \mu_{1,0} = \Delta\mu_{mix} + \Delta\mu_{elastic} + \Delta\mu_{ionic} \quad \text{Equation 1}$$

In which, μ_1 is the chemical potential of the solvent in the polymeric gel, $\mu_{1,0}$ is the chemical potential of the pure solvent, $\Delta\mu_{mix}$ and $\Delta\mu_{elastic}$ are the mixing and elastic contributions to the total change in chemical potential, respectively. $\Delta\mu_{ionic}$ is the contribution of the chemical potential change due to the ionic character of the hydrogel.

By inspection of the total chemical potential equation (Equation 1), if we are able to maintain or minimize changes to the structural crosslinking parameters of the network so that

$\Delta\mu_{elastic}$ is not significantly affected, as well as maintain temperature and solvent type, we expect an increase in the total osmotic driving force due to the presence of dissociated ionic charges in the hydrogel structure, thus increasing its water removal capacity, due to strong ion-dipole interaction. Thus, due to its improved water absorption characteristics and dominant share in polyacrylamide market [44], anionic polyacrylamide such as (poly(AAm-co-SA)) has shown to be a good candidate for hydrogel synthesis with improved water retention performance at a reasonable cost.

Studies that applied sodium acrylate hydrogels in their pure form (sodium polyacrylate hydrogels), or copolymerized with acrylamide (poly(AAm-co-SA) hydrogels), and compared them with other synthetic polymers have demonstrated that the efficiency of the process of removing water from fuels is in fact considerably increased, as predicted by thermodynamics. Under the same process conditions, Gonçalves et al., 2021 [37] showed that poly(AAm-co-SA) hydrogel was able to remove 45% more water from saturated biodiesel than neutral polyacrylamide hydrogel and 27% more than poly(AAm-co-AA) hydrogel. In the same study, water contents of about 860 ppm were observed in biodiesel (initially at 1,514 ppm) treated with poly(AAm-co-SA) hydrogel, applying a ratio of 8 mg hydrogel/mL of fuel; whereas, to achieve similar performances using neutral polyacrylamide and poly(AAm-co-AA) hydrogels, around 370 mg/ml and 304 mg/ml were needed, respectively.

In addition to increasing the water removal capacity, the poly(AAm-co-SA) hydrogel have showed water removal kinetic about twice as fast as the poly(AAm-co-AA) hydrogel when immersed in distilled water for 4 hours [26].

3.3.2 *Hydrogel mass/fuel volume ratio effect on water removal efficiency*

Gonçalves et al., 2021b demonstrated that by increasing the poly(AAm-co-SA) hydrogel/biodiesel ratio from 8 to 130 mg/ml, the water content in biodiesel is reduced from 860 ppm to 453 ppm at room temperature ($\sim 20^{\circ}\text{C}$), achieving a lower content than that required by ASTM for biodiesel. This result is comparable with the results achieved using more mature processes such as vacuum flash distillation [49]. However, hydrogel-based process consumes less energy [37]. High mass/fuel ratios could be verified when a fluid moves through a packed-bed column. Polyacrylamide and poly(AAm-co-AA) hydrogels were also evaluated by the authors, and they were not able to reach the specification even at very high hydrogel/biodiesel ratios, showing once more the better performance of sodium acrylate-based hydrogels for removing water from biodiesel.

3.3.3 *Temperature effect on water removal efficiency*

Studies that applied hydrogels to remove dissolved water from biodiesel have shown that rising temperature result in greater removal kinetics and water holding capacity at equilibrium. An increase of about 20% in water removal capacity of poly(AAm-co-AA) and poly(AAm-co-SA) hydrogels at 40°C were obtained when compared to the same test performed at 25°C [45]. Efficiencies of up to 80% were achieved at the highest temperature, reaching a concentration of approximately 330 ppm, which is below the limits established by ASTM and ANP Resolution (for distributors). Similar behavior was observed using poly(AAm-co-AN) hydrogels, reaching a water content in biodiesel of 320 ppm at 40°C against 502 ppm at 25°C [28].

With the increase in temperature, the viscosity of the fluid is reduced, which provides greater wettability of the hydrogel by the fuel and, therefore, a better contact is established. Higher temperatures also contribute to the relaxation of polymer chains, making them softer and more flexible, increasing the elasticity of the hydrogel network structure. As a consequence of the superior expansion capacity of polymeric chains, more water can be held in their polymer network. All these factors for allow higher incorporation of water into hydrogels network at higher temperatures.

3.3.4 *High cross-linking density as means for mechanical stable hydrogels*

The synthesized hydrogels are generally dry, glassy in appearance and very rigid. However, when the hydrogels are immersed in an aqueous medium and they swell, a loss of mechanical strength is observed as they become soft and flexible. Hydrogels with good mechanical strength are required for industrial purification, where they must withstand to the pressure exerted by the column of liquid and to the high shear rates resulting from high operating flow rates. Reducing the distance between cross-linking points, or equivalently increasing the density of the crosslinking, will increase the strength of the material, while simultaneously offering greater resistive force to the deformation of the chain for water retention. Consequently, highly cross-linked polymer networks typically show lower equilibrium water absorption capacities. Thus, although high water absorption capacities are extremely necessary for good performance in fuel purification, these materials must still present compatible mechanical strength for this application.

Fregolente and Maciel, 2012 [31] have demonstrated that with increasing acrylamide monomer (AAm) content (2.0 to 8.0% by mass) combined with higher amounts of the cross-linking agent N,N'-methylenebisacrylamide (MBBAm) (1.0 to 4.0% by mass, in relation to the mass of acrylamide), there are more points of contact between the main chain and the cross-

linking agent and, consequently, an increase in the cross-link density, resulting in a strong but fragile hydrogel—with high modulus of elasticity, albeit brittle—with a low water absorption capacity of $W = 9.16$ g water/g hydrogel. Hydrogels synthesized with the same ratio of monomer/cross-linking agent 2:1, with half the concentration of these species in the polymerization reaction (2.0% mass of AAm | 1.0% mass of MBBAm), did not exhibit sufficient handling resistance characteristics due to lower amounts of cross-linking points in their structures, despite having a water absorption capacity of 25.5 g water/g. Hydrogels with 4.0 wt% of AAm, combined with 3.25 wt% of MBBAm, showed sufficient water absorption capacity and cross-link density characteristics for its application, and they were able to reduce the biodiesel water content by 63.6%, from 2,200 ppm to 800 ppm.

3.4 Reuse of hydrogels for removing water from fuels

The reusability of hydrogels is an important parameter for the economic viability of their application on an industrial scale, due to their competitiveness against existing dewatering technologies. These polymeric gels have proven capable of maintaining the same performance of water removal from fuels after several reuse cycles, whether they are regenerated or not. Usually, the hydrogel regeneration process consists of immersing the material in a desiccant fluid such as ethanol followed by drying.

Li et al., (2021) [27] reused poly(N-isopropyl acrylamide) (PNIPAm) and polycationic (N,N'-dimethylaminopropyl acrylamide) (PDMAPAA) hydrogels, without regeneration, to remove dissolved water from jet fuel for several cycles in batch runs. After each cycle, the hydrogels were removed from the fuel and immediately placed in another flask containing untreated fuel. The performance of these reused hydrogels remained stable and they were able to reduce the water content from 45 ppm to ~7 ppm, even after seven cycles. Also in non-continuous tests, regenerated poly(AAm-co-SA) hydrogels applied to remove water from marine diesel oil (MDO) statistically maintained the same removal efficiency after four reuse cycles[13].

In continuous processes, similar performances were obtained when employing never-used and regenerated sodium polyacrylate hydrogels filled in a fixed bed to treat cloudy diesel [35]. The two hydrogels efficiently diminished the turbidity of diesel oil from 16.4 NTU at the inlet to < 3 NTU at the outlet, appearing completely clean at end of the columns as shown in Figure 2.

4. Conclusions

Synthetic hydrogels have been shown to be highly promising for dehydration of liquid fuels. The selection of hydrogels for this application depends mainly on the chemical

characteristics of the fuel, especially its polarity, and the required level of removal. Petroleum-based fuels such as diesel have a lower affinity for water and it can be efficiently removed by hydrogels in both batch and continuous processes, to a level below that required by international standard specifications. For purification of hydrophilic fuels, such as biodiesel and its diesel blends, ionic charged hydrogels have been shown to be essential to increase the osmotic strength of the system and provide greater water removal. Hydrogels with ionic charges combined with moderate ratios of hydrogel mass/fuel volume and temperature conditions provide a route to obtain water content in biodiesel within the level set by the regulatory agencies.

The new hydrogel technology to remove water from fuels has several advantages when compared to other commercially available technologies. They offer a complete solution since they remove free and dissolved water, they are simple to operate, and can be reused. Moreover, the hydrogel technology presents as a differential its application in a wide range of scales: industrial processes, distribution centers, gas stations, or in vehicle filters.

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