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Chapter

Potential of Lignin Valorization with Emphasis on Bioepoxy Production

Mohamed H. El-Moayed, Justin Kühn, Sea-Hyun Lee, Mahmoud Farag and Sherif Mehanny

Abstract

Lignin is the second most abundant natural polymer after cellulose. It has high molecular weight and poor dispersity, which lowers its compatibility with other polymeric materials. Accordingly, it is hard to integrate lignin into polymer-based applications in its native form. Recently, lignin valorization, which aims to boost lignin value and reactivity with other materials, has captured the interest of many researchers. The volatility of oil and gas prices is one strong incentive for them to consider lignin as a potential replacement for many petroleum-based materials. In this chapter, lignin valorization processes, namely hydrogenolysis, pyrolysis, hydro-thermal liquefaction, and hydro-thermal carbonization, are discussed in brief. The chapter also discusses the synthesis of lignin-based epoxy resin as an already existing example of a lignin-based product.

Keywords: lignocellulose, valorization, biomass, waste management, bioresins, eco-friendly

1. Introduction

Environmental friendliness and green alternatives have been a significant concern, to reduce carbon footprint and alleviate environmental toxicity problems [1, 2]. Lignocellulose is the most abundant renewable biopolymer ever existing [1, 3]. Cellulose, hemicellulose, and lignin count up to more than 90–95% of lignocellulosic texture [4]. Lignin is an organic polymer whose structure is highly dependent on its native source. In general, it is an amorphous, isotropic material, covered, for example, in the wooden stem of a plant. All plants and algae comprise lignin in different amounts, in addition to cellulose and hemicellulose [5, 6]. Lignin amount also differs according to the time of harvesting. The lignin content gets much higher, the older the plant gets. For instance, lignin presents about 12.4 ~ 29.4% of hemp [7] and 2.5 ~ 3.8% of flax [8]. Nevertheless, the highest lignin content can be found in woods, with a percentage of 20 ~ 30% [9]. Lignin leads to a so-called lignification of the stem (from Latin: *lignum*). This makes it more robust and stronger by gumming the cellulose part together [10]. This partly also

occurs in leaves. It is extracted from these natural sources via physical, chemical, or biological methods.

Physical processes include steam explosion and mechanical grinding. The physical route yields high-purity lignin. However, it is hard to be industrially upscaled. Chemical processes commonly used for pulp and paper, such as the kraft and sulfite process, utilize reagents that trigger reactions, yielding moderate-purity lignin, depending on the parameters. The harsher the reaction environment and chemicals utilized, the lower the quality of lignin will become. New processes such as organosolv process running on water and ethanol as main solvents with an optional acid catalyst will result in higher quality lignin due to the mild reaction conditions. However, to obtain high-purity lignin, the respective fraction will need to undergo further treatment. Finally, the biological option involves enzymes that break lignin bonds with cellulose and hemicellulose. Despite producing high-purity lignin, this technique is not favorable due to its low speed. The majority of industrial lignins are extracted chemically from their sources.

Extracted lignin has high molecular weight and poor reactivity with other polymeric materials. Hence, its uses are limited to combustion applications. Around 98% of lignin is burnt as a low-value fuel, while the rest is fabricated into commercial products [11]. In order to increase lignin utility, its structure has to be modified first. The purpose of the modification is to enhance lignin reactivity and homogeneity and lower the probability of infusible solids formation. Boosting lignin value is called “lignin valorization.” **Table 1** summarizes different valorization processes, which are covered in the following section.

With a share of 4.3% of the European pulp production and total production of 5 million tonnes of paper in Austria, there is an approximate market demand of 116.3 billion tonnes of pulp only in Europe. Multiplying this with a mean value of the content of lignin in wood, taking 25%, there is a total yearly amount of 29 million tonnes of lignin. Adding further lignin sources as mentioned above as well, the value is significantly increasing. The following chapter addresses, how the valorization process takes place to not waste this amount of lignin. Afterward, Chapter 3 deals

Process	Temperature range, °C	Atmosphere and media	Catalyst	Main products	Reference
Hydrogenolysis	40–200	H ₂ water (or alcohol)	Supported transition metal	Mixed aromatics	[12]
Pyrolysis	500–1000	N ₂ , CO ₂	Acid, alkali, metal, metal oxide	Pyrochar and bio-oil	[13]
Hydrothermal liquefaction	200–400	N ₂ , CO ₂ Water (or Organic Solvent)	Acid, alkali, metal, metal oxide	Bio-oil and phenolics	[14]
Hydrothermal carbonization	160–240	N ₂ water (or organic solvent)	Acid, alkali	Hydrochar	[15]

Table 1.
Lignin valorization processes.

with further processing of lignin-based epoxy resin synthesis. Concluding, a short chapter will summarize the findings.

2. Lignin valorization processes

To describe how to use lignin as a valuable resource (see **Figure 1**), this chapter initially describes the hydrogenolysis of lignin. Afterward, the pyrolysis and gasification are described that lignin might be used as an energy source. Material collection on a wet basis is discussed in the chapter on hydrothermal liquefaction (HTL). This chapter closes with a discussion of hydrothermal carbonization (HTC) of lignin.

2.1 Lignin hydrogenolysis

Lignin hydrogenolysis is a three-step process. First, lignin macromolecule functional groups are reduced without breaking the main structure. Second, macromolecules are broken into phenolics and arenes. Third, further reduction of second-step molecules into alkanes takes place.

2.2 Lignin pyrolysis and gasification

Lignin pyrolysis is an oxygen-free thermochemical conversion process. It aims to recover energy and other materials under a temperature ranging from 300 to 800°C [16]. Typical products of pyrolysis are biochar, bio-oil, and syngas. The quality of these products is highly dependent on temperature and dwell time. Longer residence time, or slower heating rate, reduces the bio-oil yield and boosts syngas content due to secondary cracking [17]. Lignin molecular weight is another important factor. Low molecular weight leads to the formation of CH₄, CO, and CO₂ from the methoxy group, whereas high molecular weight yields guaiacol and alkyl guaiacol [18].

While lignin pyrolysis is working in an oxygen-free environment for the production of syn-gas as the main product, which could also be of interest for future industries, as over 50% of syn-gas production is still petro-based and is based on the injection of pure oxygen or steam for stoichiometric balance, crucial for the

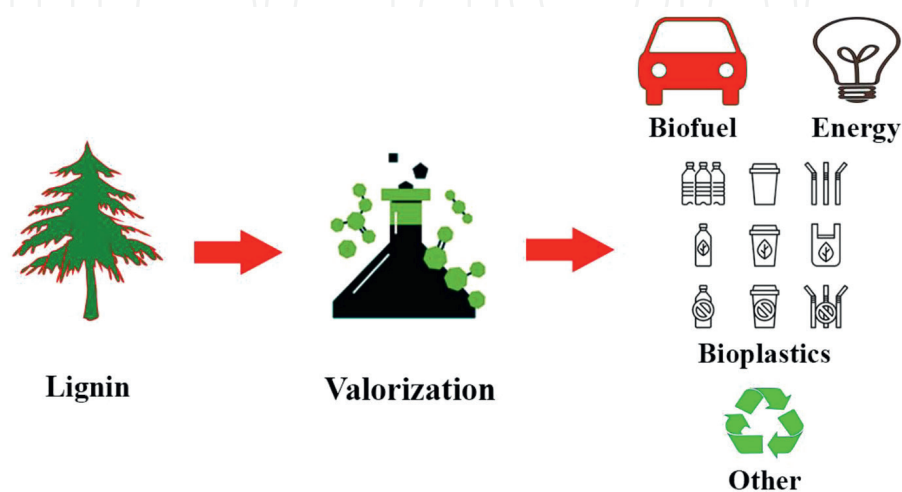


Figure 1.
Lignin valorization process.

production of higher quality syn-gas from biomass. The constitution of syn-gas is highly dependent on the biomass utilized, gasifier type, and agent, as well as operational conditions. It should be noted that lignin in itself is limited in hydrogen that is covalently bound to the structure ranging in the region of 5–6 wt%, which would be the maximum theoretically achievable ceiling, with respect to the energetic input needed for extraction. With gasification utilizing steam, the hydrogen output at higher temperatures is influenced due to the reaction between water and free carbon in the reaction chamber. Currently, the gasification of wood as the main feedstock is fairly common. However, the true interesting contender is biomass based on agricultural residues, such as straw, husks, shells, and more, which exhibit comparable values in syngas composition from the current benchmark in lab scale [19, 20].

2.3 Lignin hydrothermal liquefaction, HTL

Recently, HTL has been extensively studied as a biomass conversion method with water as a reaction medium. HTL is an efficient process of lignin transformation during which pyrolysis and hydrolysis take place simultaneously [14]. HTL is considered an eco-friendly and sustainable technology with the advantages of short residence time, high conversion rate, and less pollution.

Although lignin is abundant in plethoric quantities naturally, HTL has not reached the industrial scale, not even the pilot level because of troublesome depolymerization and separation. In contrast, lab-scale studies have been successful in lignin transformation in which 60 wt% low-weight aromatic molecules were obtained from oxidized lignin [21].

2.4 Lignin hydrothermal carbonization, HTC

HTC is an eco-friendly, convenient, and low-cost method [22]. Comparably, HTC offers distinctive advantages, which are not limited to no need for pre-drying, low processing temperature (180–350°C), avoiding air-polluting nitrogen oxides, and sulfur oxides dissolution in water [23]. During HTC operation, lignin is heated in a high-pressure autoclave in the presence of subcritical water and pressure ranging between 2 and 10 MPa. Reaction mechanisms comprise hydrolysis, dehydration, recondensation, decarboxylation, and aromatization [15], which are collaborative and concurrent. Similar to pyrolysis, key roles are played by the reaction temperature, residence time, and source of raw materials in the HTC process that yields hydrochar [24].

3. Lignin-based epoxy resin synthesis

Epoxy resins were first introduced in Europe by P. Schlack in 1939 [25]. They are a group of thermosetting resins (see **Figure 1**) that require curing to harden. The ratio of the curing agent, or the hardener, to the resin affects the overall performance of the polymer [26]. In general, epoxy possesses excellent thermal and mechanical properties. Hence, it is favorable in the fields of coating, electronic packaging, and thermal insulation [27], to name a few.

One of the most common epoxy resins is the diglycidyl ether of bisphenol A (DGEBA). DGEBA is the product of an epichlorohydrin (ECH) reaction with bisphenol A (BPA) in the presence of a basic catalyst [26]. Since BPA is a petroleum-based chemical, DGEBA is not biodegradable, thereby having a great potential to damage

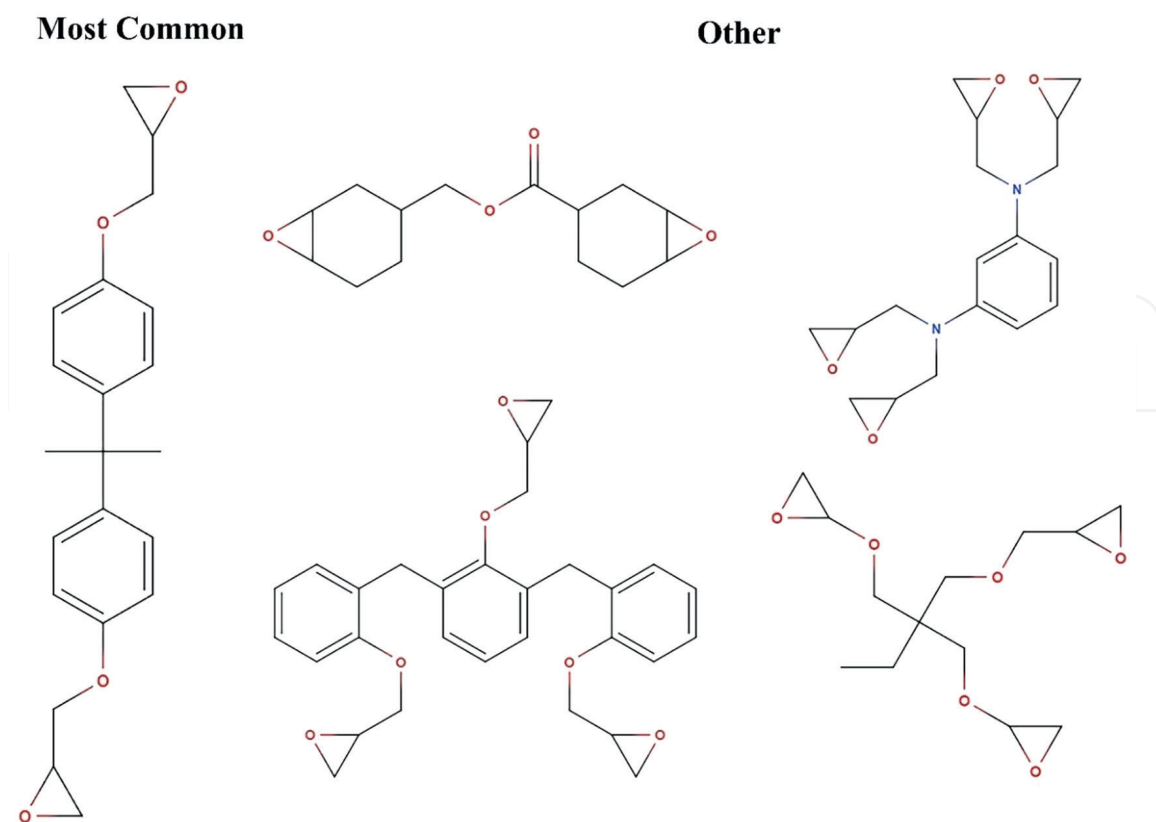


Figure 2.
Different types of epoxy resins.

the environment [28]. Being an environmental hazard has never been enough of an incentive to search for safer options. However, the recent volatility of oil and gas prices adds to the list of reasons that justify those options [29].

Lignin is a natural polymer whose structure is a complex network of phenylpropane units, namely *p*-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol [29, 30]. **Figure 2** shows the three building blocks of lignin. Lignin structure is very similar to that of BPA, which qualifies the former to be an excellent substitute for the latter [28]. Luckily, lignin is the second most abundant macromolecule after cellulose. Therefore, it is no surprise that researchers find lignin-based epoxy resins worthy of their attention. By far, there are three methods to integrate lignin into epoxy resin synthesis. They are a) physical blending of lignin and epoxy resin, b) epoxidation of lignin after pretreatment, and c) epoxidation of unmodified lignin [31, 32].

3.1 Physical blending of lignin and epoxy resin

In this method, lignin is blended with petroleum-based epoxy resin to form a binary mixture, which is then cured at a proper temperature. *Simionescu et al.* [33] stated that epoxy resins with 25 ~ 50 wt% lignin exhibited good mechanical and dielectric properties after curing at elevated temperatures. In a different study, *Behin et al.* [34] reported that adding small amounts (< 2.5 wt%) of kraft lignin and Sal-A nanoparticles to uncured epoxy resins boosted their overall mechanical performance after cross-linking. They also mentioned that the additives had a positive impact on the curing reaction. In particular, both peak temperature and total heat of the curing reaction dropped significantly.

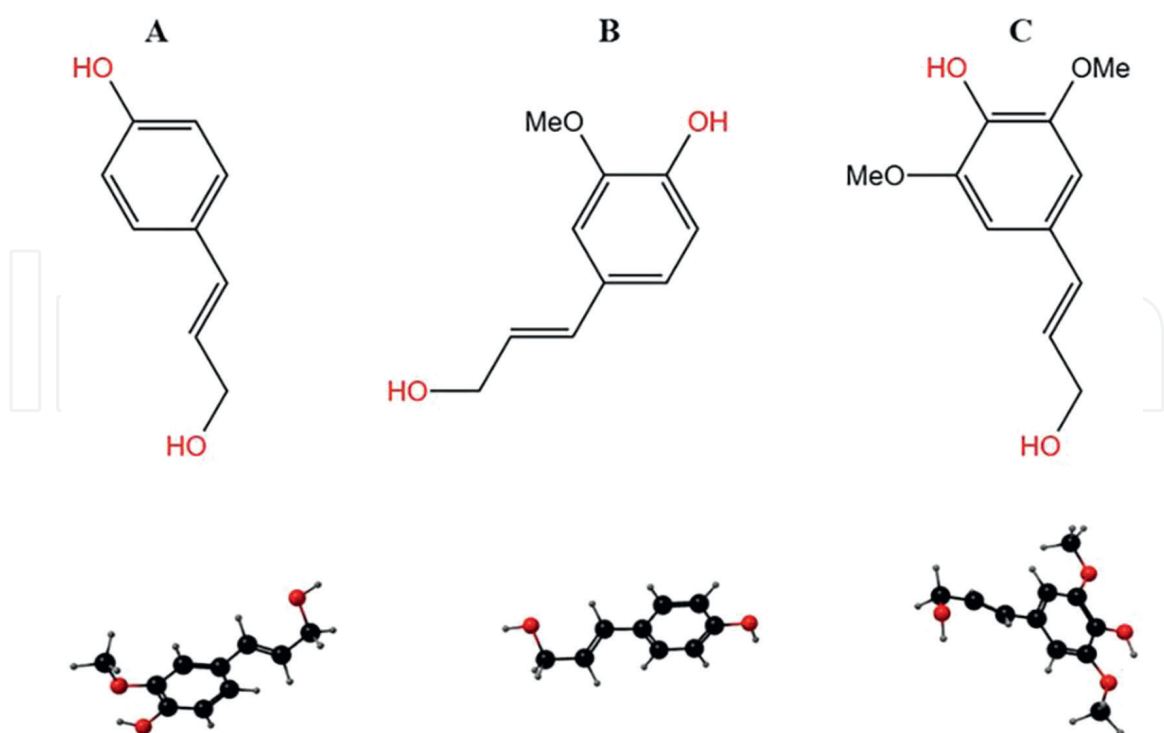


Figure 3.
Lignin's basic components: *p*-coumaryl alcohol (A), coniferyl alcohol (B), and sinapyl alcohol (C).

Pan *et al.* [35] considered the addition of lignin as a cross-linker. They mixed aminated lignin (see **Figure 3**) with a regular curing agent (W93) at 80°C. Then, a proper amount of liquid epoxy was added to the blend after cooling to room temperature. The epoxy-hardener mixture was cured in a baking oven afterward. Results showed that the thermal behavior of epoxy gradually improved with increasing the amount of aminated lignin in the hardener system up to 50%. Further increase in lignin content caused a drastic deterioration in performance, owing to agglomeration.

In light of the abovementioned studies, it is clear that the simple blending approach is straightforward and versatile. However, owing to lignin heterogeneity (steric-hindrance effect), the complete substitution of petroleum-based materials is not feasible with this technique.

3.2 Epoxidation of lignin after pretreatment

Large molecular weight and poor dispersity lower lignin compatibility with polymeric compounds. Better reactivity can be realized via chemical modification, for example, phenolation, hydroxymethylation, and demethylation. For instance, Zhang *et al.* [36] synthesized phenolated lignin, hydroxymethylated lignin, and demethylated lignin from native lignin through chemical modification. The modified lignin types were reacted with epichlorohydrin to obtain lignin-based epoxy resins, which were then utilized as feedstocks for new grouting materials. The newly prepared lignin-epoxy grouts outperformed the commercial ones, displaying great potential in replacing petroleum-based materials.

Zhen *et al.* [28] proposed a novel strategy (see **Figure 4**) to produce lignin-based epoxy resin from phenolated lignin. They demonstrated in their work the superiority of their new approach over the traditional one. They noticed a slight deterioration

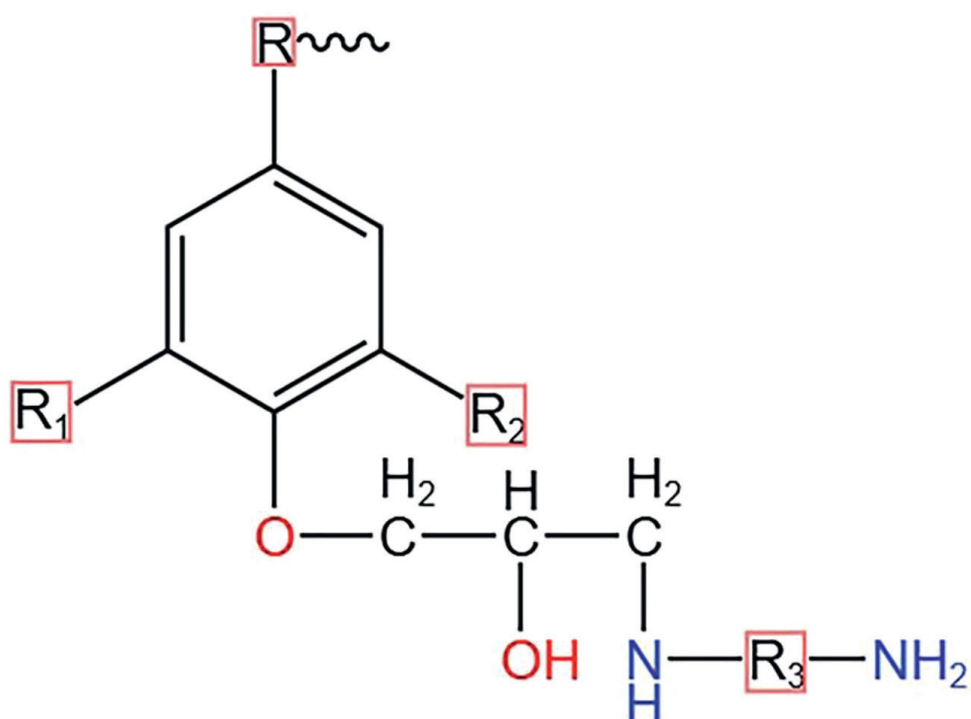


Figure 4.
 Aminated lignin structure.

in thermal-mechanical performance when the lignin amount exceeded 20% (80% phenol). However, the values were still better than lignin-free resins (**Figure 5**).

Similar to the first approach, the steric-hindrance effect of lignin is still inevitable. However, its impact is not as bad as in the simple blending approach. With the technique discussed herein, the complete substitution of petroleum-based materials is achievable, which is a major step in the right direction toward the production of petroleum-free thermosets.

3.3 Epoxidation of unmodified lignin

Unmodified, or technical, lignins refer to a large group of lignin by-products whose properties are very different from their native form [37]. Kraft lignin, lignosulfonate lignin, and organosolv lignin are some good examples of unmodified lignins [38].

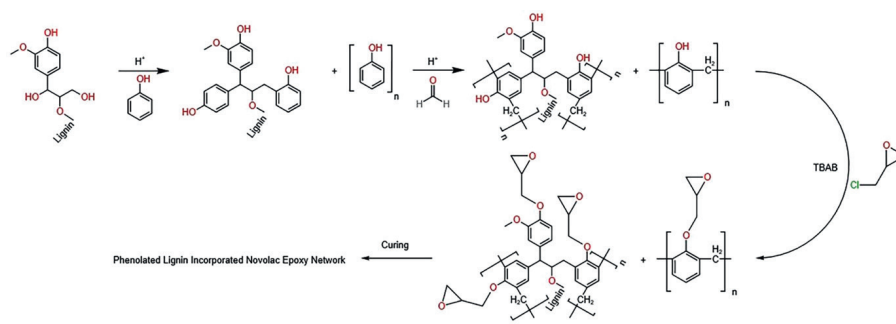


Figure 5.
 PLIEN production process as proposed by Zhen et al. [28].

Like the previous method, BPA can be completely replaced with unmodified lignins in epoxy resin formulation. The main difference between the two techniques is the cost. Since technical lignins are already available as industrial wastes, no additional processing cost is added, which qualifies the current method for large-scale production [32].

4. Conclusions

The current chapter reviewed the current state of the art in lignin valorization starting from the extraction process to utilization via lignin-based epoxy resin synthesis. The key conclusions are as follows:

- While about every plant-based source has lignin as a base building material, the constitution will vary greatly depending on the type utilized.
- Lignin is extracted from the source either physically, chemically, or biologically and varies in quality depending on the technology.
- The greatest majority of industrial lignins are chemically extracted from their native sources.
- Lignin described through their bond type or content of S-, P-, and C- type alcohols, due to their complexity, contains various chemical groups interesting for a wide array of industries.
- Lignin cannot be utilized in its original form; due to its heterogeneity and impurities from the treatment of the source material, however, it can be valorized through fractionation to enhance its homogeneity.
- Hydrogenolysis, pyrolysis, HTL, and HTC are examples of lignin valorization processes.
- There are three synthesis techniques to produce lignin-based epoxy resins. They are as follows: (a) simple blending of lignin and petroleum-based epoxy, (b) epoxidation of modified lignin, and (c) epoxidation of unmodified lignin.
- In the simple blending approach, lignin can replace a small percentage of petroleum-based BPA. In contrast, the other two techniques can achieve complete substitution of BPA.

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