## Chapter 2

# Flammability performance of biocomposites

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## 2.1 INTRODUCTION

Bio-based composites are witnessing a growth in different industrial sectors, mainly ranging from construction to aerospace industries. This recent surge in the use of biocomposites is motivated by factors such as depletion of and high cost of petroleum reserves, comparable technical properties of biocomposites, and the current shift toward environmentally friendly and sustainable materials which in turn is supported by the implementation of environmental legislation such as REACH Act (Registration, Evaluation, Authorization and Restriction of Chemical substances) and other bio-based incentives.

Bio-based products have been mainly used as flexible and rigid packaging materials where stringent flammability measures did not always have to be met. As recent trends seem to dictate the growing use of biopolymers [like polylactic acid (PLA)] and biocomposites in the transportation and electronics sector, it has become very relevant to formulate strategies to improve flammability by the use of flame retardants to meet the various industrial regulatory standards of these sectors. A few examples of such regulatory measures include for aerospace applications; components used as interior panels must exhibit heat release rates (total heat release and peak heat release)  $\leq 65 \text{ kW/m}^2$  when tested on a Ohio State University heat release apparatus. For the automotive sector, the Federal Motor Vehicle Safety standard 302 stipulates that horizontal burn rate of the samples must not exceed 102 mm/min.

There are four main families of flame retardant (FR) chemicals: inorganic, organophosphorus, nitrogen-based, and halogenated. There are synergists that act in combination with the FRs (e.g., antimony trioxide, zinc borate). Many of the traditional FRs, however, pose environmental/ecotoxicological threats, whether it is during manufacture, application, or combustion, or at end-of-life disposal. For example, antimony trioxide is a possible carcinogen and some of brominated and chlorinated FRs may form dioxins and furans on combustion or can be persistent, bioaccumulative, and/or toxic to humans, and this has led to a market call for halogen-free FRs There is growing evidence that these FRs can accumulate in people and cause adverse health effects-hormonal imbalances, interference with reproductive systems, thyroid and metabolic function, and neurological development in infants and children-and this has resulted in restrictions like REACH being imposed. The full-scale implementation of REACH will affect more than 3000 chemicals across a huge range of industrial and consumer products. Another legislative measure-the Stockholm Convention on Persistent Organic Pollutants (POPs)-has banned the production and use of 21 chemicals of which four are brominated FRs.

Consequently, new ways are being sought to find more environmentally friendly FRs or fire resistance systems. These include modifications to new inorganic-based FRs, the use of nanocomposites (e.g., nanoclays), and nontoxic "green-chemistry" solutions. Also, in the use of green/environmentally friendly FRs the biodegradable nature of the biopolymers is retained.

## 2.2 FLAMMABILITY TESTING TECHNIQUES

#### 2.2.1 Cone calorimetry

Cone calorimetry is one of the most common fire behavior tests used to study the rate of heat released by materials exposed to radiant heat flux. Its principle is based on the measurement of decreasing oxygen concentration in the combustion gases of the sample that is subjected to a given heat flux (10-100 kW)

m<sup>2</sup>). The analysis of combustion products and gaseous species can be performed by installation of a Fourier transform infrared (FTIR) spectrometer [1].

The most important parameter obtained from cone calorimetric testing is the heat released per unit of time and surface area (heat release rate, HRR) expressed in kW m<sup>-2</sup>. The fire properties of materials are assessed by studying the gradual development of HRR over time, that is, the value of its peak maximum (PHRR) or HRR maximum. The calculation of HRR is based on Huggett's study on the estimation of HRR via oxygen consumption measurements [1a]. This study concluded that most organic materials release an amount of heat that is practically proportional to the amount of oxygen consumed while burning. The proportionality factor is constant from one material to another and is equal to 13.1 kJ g<sup>-1</sup> consumed oxygen, with an accuracy of approximately 5%. The total heat release (THR) [2] is obtained by integration of the HRR versus time curve expressed in MJ m<sup>-2</sup>. Cone calorimetry, in addition to the aforementioned parameters, also provides information on time to ignition (TTI), time to flame out (TFO), mass loss rate, levels of oxygen, carbon monoxide, and carbon dioxide, and total smoke released (TSR). Furthermore, it provides more detailed fire characteristics, with HRR being the most widely used parameter for evaluating the fire properties.

## 2.2.2 Pyrolysis combustion flow calorimetry

Pyrolysis combustion flow calorimetry (PCFC) is a fire test method for evaluating the combustibility of milligram-sized samples [3]. It is also known as microscale combustion calorimetry (MCC) and is now a standardized technique classified as ASTM D7309-07. PCFC reproduces the solid state and gas phase processes of flaming combustion in a nonflaming test. This is achieved by controlled pyrolysis of the sample in an inert gas stream followed by high temperature oxidation of the volatile pyrolysis products. The heat of combustion of the pyrolysis products is measured by the use of oxygen consumption calorimetry. This method combines the constant heating rate and flow characteristics of thermal analysis (i.e., thermogravimetry) with the ability to determine the heat of combustion typical of oxygen bomb calorimetry. PCFC determines the heat release and heat release rate using an oxygen consumption method.

The heat release capacity is the maximum potential of the material to release combustion heat in a fire. From the above derivation, the heat release capacity ( $\eta c$ ) is a combination of material properties, and consequently, is itself a material property as measured in PCFC. The use of Heat release capacity (HRC) as a measure of fire risk is based on the assumption that the maximum specific heat release rate at the decomposition temperature reached at constant heating rate correlates with the mass loss rate during pyrolysis in a fire characterized by a transient temperature gradient [4].

#### 2.2.3 Limiting oxygen index

Limiting oxygen index (LOI) is the minimum concentration of oxygen in a mixture of oxygen and nitrogen that is needed to support the flaming combustion of a material. It is expressed in volume percent (vol%). It was first introduced in 1966 by Fenimore and Martin and is used to indicate the relative flammability of materials. It is standardized in the United States (ASTM D 2863) and in France (NF T 51-071), as well as internationally (ISO 4589). The method involves placing a sample vertically within a controlled atmosphere and its top inflamed with a burner. LOI, the minimum concentration of oxygen in a mixture of oxygen and nitrogen that either maintains flame combustion of a material for 3 min or consumes a length of 5 cm of a sample, is expressed as:

$$LOI = 100 \times [O_2] ([O_2] + [N_2])$$

$$(2.1)$$

where  $[O_2]$  and  $[N_2]$  are the concentrations of oxygen and nitrogen gases, respectively.

Materials with LOI values less than 21% are classified as combustible, but those with LOI greater than 21 are classed as self-extinguishing since their combustion cannot be sustained at ambient temperature without an external energy contribution. Materials with a high LOI value generally exhibit a better FR property. This method remains one of the most important screening and quality control tools in the plastics industry to characterize both the ignitability and flammability resistance. However, LOI measurements are taken at room temperature and LOI values decrease as temperature increases. This means that self-extinguishing cannot be considered a fast rule, since materials with high LOI values at room temperature may burn without self-extinguishing under intense fire conditions. It should be noted that melting and dripping of a polymer during the LOI test may cause a specimen to extinguish and thus give misleading high LOI values. In addition, LOI has a limitation as a test for nanocomposites in that organomodified nanoclay-reinforced polymer nanocomposites exhibit decreased LOI values due to an increased flame spread rate over the surface of the specimen, while inside the material nanoclays form a barrier layer that limits the propagation of fire [5]. The advantages of LOI are that it provides a convenient, reproducible means of determining a numerical measure of flammability. Furthermore, the test equipment is inexpensive and only a small sample size is required for testing.

## 2.2.4 Underwriters laboratories 94 (UL94)

UL 94 is a set of tests approved by Underwriters Laboratories Inc. as flame tests for plastics materials for parts in devices and appliances. It includes a range of tests such as small and large flame vertical (V) tests, horizontal (H)

tests for bulk and foamed materials, as well as radiant panel flame spread test. The commonly used test is UL 94V in terms of practice and usage. It measures ignitability and flame spread of vertical bulk materials exposed to small flame. It is equivalent to international standard IEC 60695-11-10 (Test method B) for small flames (50W) and ASTM D3801-10 [6]. The UL 94 test is widely used both in industry and academic research centers. It is intended to meet industrial requirements, as well as classify polymeric materials hierarchically. The information obtained remains limited due to its basic and unrefined character.

## 2.2.5 Ohio State University heat release apparatus (OSU)

The OSU is a technique used to measure the RHR of materials and products in a forced flaming combustion. Both heat and smoke release are measured from the moment the specimen is injected into a controlled exposure chamber. Measurements are continued during the period of ignition and to such a time that the test is terminated. Although the OSU heat release apparatus is a good tool for flammability testing, there are known limitations of this test method [7]. The heat and smoke release depend on several factors such as formation of surface char, formation of adherent ash, sample thickness, and method of mounting. Heat release values are a function of the exposed tested area of the specimen. The test method is restricted to the specified specimen size of materials, products, or assemblies. In the case of products, the test specimen (i.e., prototype) is representative of the product in actual size. At very high specimen HRR, flaming above the stack is possible making the test invalid.

There is no established general relationship between HRR values obtained from horizontally and vertically oriented specimens. Specimens that melt and drip in a vertical position are tested horizontally and hence vertical testing remains a problem for testing thermoplastic materials in the OSU.

## 2.3 CASE STUDIES

#### 2.3 1 Biopolymers and biocomposites

The different types of FRs and their use in natural fibers and natural fiber–reinforced composites has been documented in detail by Mngomezulu and John [8]. Generally it has been seen that the methods of imparting fire resistance in natural fiber–reinforced composites has been through three main routes [9]:

- Chemically treating the natural fibers with suitable FRs;
- Treating the matrix (petroleum-based/biopolymer) with suitable FR additives during melt compounding stage;
- Use of FR coatings on the molded natural fiber composite

The choice of the FR depends upon the type of matrix polymer and processing technique used. In the case of natural fibers, commonly used FRs include phosphorus and nitrogen-based systems, which function in the condensed phase and promote char formation by converting fiber structure into char and reduce fuel formation. The most commonly used FRs for thermoplastic polymers are metal hydroxides (aluminium hydroxide/magnesium hydroxide), expandable graphite, ammonium polyphosphate, intumescent systems, and nanoclays. Metal hydroxides act by undergoing exothermic reactions at elevated temperatures releasing vapor which retards the fire-forming reactions. Metal hydroxides need to be used at high loadings and usually has an adverse effect on mechanical properties. Phosphate-based systems interfere with polymer decomposition and promote char formation. FR coatings act as a barrier coating and are more often ceramic or intumescent-based. Intumescent coatings upon exposure to fire form a carbonaceous layer which acts as a thermal barrier and protects the underlying polymer system.

In recent times, the use of nanofillers in PLA has been of particular interest as researchers have found that such nanocomposites exhibit improved mechanical properties at low loadings (3%-5%) along with increased flame resistance [10]. Addition of clay has been shown to reduce the peak heat release rate (PHRR) for PLA-based nanocomposites [11]. A decrease or increase in smoke production has also been observed. It has been proposed that when the polymer matrix is exposed to heat, the clay platelets migrate to the surface providing a protective layer that delays gasification of the layer below. The clays also form a carbonaceous-silicate char which acts as a heat barrier, increasing thermal stability, and as a physical barrier to mass transport of oxygen and volatile products.

Solarski et al. investigated the flammability properties of melt-spun PLA/ montmorillonite multifilament nanocomposite fibers using the cone calorimeter [12]. They found a large (38%) decrease in the PHRR of the nanocomposites when compared to the pure PLA. Tensile strength and Young's modulus, however, decreased upon addition of clay. In addition to clay, multiwalled carbon nanotubes (MWCNTs) have also been used in PLA but limited enhancement was observed as MWCNTs were not uniformly dispersed in PLA [13]. Another nanofiller that has potential is expanded graphite (EG) which when incorporated in PLA reduced the PHRR by 30% when compared to virgin PLA [14]. The use of natural halloysite as effective green FRRRs for thermoplastic compounds was presented by Dharia and Zetoun [15].

Although PLA nanocomposites exhibit lower flammability properties in terms of cone calorimetry, they often fail in other flammability tests (limiting oxygen index and UL-94) [16]. The fire resistance of the nanocomposite can, however, be improved by combining the nanofiller with conventional FRs. When using nanofillers, the properties achieved depend not only on the prop-

erties of the nanofiller and polymer but also on interfacial characteristics and the method of preparation. Layered silicate nanocomposites, for example, result only when an intercalated or exfoliated structure is formed. An intercalated nanocomposite is one in which the polymer chains are inserted into the layered silicate structure in a crystallographically regular fashion. When the individual silicate layers are separated in a continuous polymer matrix, an exfoliated structure is obtained. When the polymer is unable to intercalate between the layered silicates, phase separation occurs and the properties remain the same as that for a traditional microcomposite. For particle- or fibrous-reinforced nanocomposites the dispersion and nanofiller-polymer interface is crucial in determining the properties of the material. Uniform dispersion is usually attained by chemical modification of nanofillers to enhance the interfacial adhesion between the nanofillers and polymer matrix. In a recent study, PLA containing melamine grafted MWCNTs were found to exhibit a 28% reduction in PHRR. This was attributed to high level of dispersion in treated MWCNTs-reinforced PLA when compared to unmodified systems. Another interesting observation was the formation of a compact and cohesive char for the functionalized MW-CNT-reinforced PLA while unmodified PLA exhibited islands of char devoid of any cohesion [17].

The use of nanoclay (C30 B) and polylactic acid grafted glycidyl methacrylate (PLA-g-GMA) on flammability properties of silane-treated banana fiber-reinforced PLA composites was investigated by Sajna et al. [18] Flammability properties measured by UL-94 horizontal burning test and cone calorimetry showed that the PHRR reduced by 22.6% in the presence of nanoclay (Fig. 2.1). This was attributed to the fact that nanoclay accelerated the char formation and hence could reduce the flammability of PLA samples.

Another group of FRs commonly used for natural fiber composites are the inorganic hydroxides. In a recent study, Mg  $(OH)_2$  and Al  $(OH)_3$  were used to improve the fire resistance of sisal [19] and saw-dust [20] –reinforced polypropylene composites. However inorganic hydroxides have been reported to reduce the mechanical properties of composites and hence the use of Ca(OH)<sub>2</sub> as a FR was studied by Zhao et al. [21]. for hemp-reinforced polypropylene composites. In this study, Ca  $(OH)_2$  was used to modify the hemp fiber surface and not incorporated in the compounding stage as in previous studies. Fig. 2.2 shows the LOI values of the composites, and it can be seen that the LOI values registered an increase indicating improved flame retardance, and this was attributed to the fact that Ca(OH)<sub>2</sub> decomposed to form CaO and water which aided in fire resistance. The authors also observed that addition of ammonium polyphosphate (APP) further improved flame retardance due to synergistic interaction between CaO and APP.

In a detailed study, Sypaseuth [22] observed that best flammability results are obtained when multicomponent FR systems are used. In the study dealing with kenaf fiber–reinforced PLA composites, the synergistic effects of multicomponent systems of expandable graphite with ammonium polyphosphate



FIGURE 2.1 HRR graphs of compatibilized bionanocomposites.



FIGURE 2.2 Limiting oxygen index of the treated and untreated hemp and their reinforced composites.

and magnesium hydroxides with layered silicates were studied. It was observed that for both systems, excellent flame retardance was obtained at low level of FRs.

In another study [23] involving synergistic systems, ammonium polyphosphate (APP) and distiller's dried grains with solubles (DDGS) as the natural charring agent was used for polylactic acid systems. The surfaces of both APP and DDGS were coated by degradable polymeric FR resorcinol di(phenyl phosphate) (RDP). The main observation of the study was that the limited oxygen index value of the biocomposites with loading of 15 wt% C—DDGS and 15 wt% C—APP reached 32.0%, and a UL—94 V—0 was attained. The biocomposites also exhibited good mechanical properties with the tensile strength of the samples being maintained at 57 MPa.

The effect of the FR APP on the properties of a range of natural fiber–reinforced polybutylene succinate composites was investigated by Dorez et al. [24]. The natural fibers chosen in this study were cellulose, hemp, flax, sugarcane, and bamboo and flammability was measured by PCFC and cone calorimetric testing. The main observations of this study were that the incorporation of fibers reduced the time to ignition in the composites but increased the char residues (Fig. 2.3), which led to reduction of PHRR values. The authors suggest that the synergistic effect of the presence of APP and natural fibers results in decrease of flammability properties.

The use of glycerol phosphate and phosphorus-silane treatment on flax fibers in flax-reinforced PLA/thermoplastic starch (TPS blends) was investigated by Bocz et al. [25]. The authors observed that the multifunctional ad-



**FIGURE 2.3** Residue of samples from cone calorimeter tests: (a) 90PBS10fl, (b) 85PBS15fl, (c) 80PBS20fl, (d) 70PBS30fl.

ditive system resulted in reduction of flammability properties by 40%. The composites also exhibited well-balanced strength and stiffness values.

In an interesting study, the effect of biochar (carbonaceous material produced when organic wastes are heated at high temperatures >500°C) on the properties of polypropylene composites was investigated by Das et al. [26]. The authors observed that the both flame retardance and mechanical properties of the composites improved with the addition of biochar. With incorporation of 35% biochar, the PHRR values were found to decrease by 54% (Fig. 2.4) and time to ignition by 43%. This was attributed to the thermally stable biochar acting as an efficient barrier to transport of oxygen and reducing the flammability.

As an extension of the above study, the synergistic effect of biochar and wool on polypropylene composites was investigated by the same authors [27]. Samples were prepared containing different ratios of biochar, wool, and APP. It was found that the incorporation of both biochar and wool fiber reduced the PHRR, smoke production as seen in Fig. 2.5 and resulted in improved LOI values. Composites containing higher amount of wool exhibited higher LOI values compared to composites containing biochar. Another interesting observation was that wool-containing composites exhibited minimal dripping of molten PP and negligible smoke production. This was attributed to the high fire resistance and char forming ability of wool fibers.

Recently the researchers at the Council for Scientific and Industrial Research (CSIR), South Africa, have been involved in the development of woven



FIGURE 2.4 Flammability properties of biocomposites (cone calorimeter).



FIGURE 2.5 Cone calorimeter results of neat PP and composites.

flax fiber–reinforced phenolic sandwich panels for use in aircraft as secondary structures. The research work involved imparting an aqueous-based phosphate FR treatment on the flax fabric to ensure that the composite panels comply with Federal Aviation Administration (FAA) and AIRBUS regulations. In addition to the primary FR, the panels contained nonfibrous natural silicate fire resistant material as well. The composite panels were reported to exhibit superior flammability, smoke, and toxicity properties for the aforementioned purposes [28].

In another study by the same group, researchers analyzed the aging and flammability behavior of woven flax fabric–reinforced phenolic composites. It was observed that the phosphate-based flame retardant treatment on the woven flax fabric reduced the PHRR, SPR, and  $CO_2$  emission rates. Biocomposites being used in the transport sector are exposed to varying conditions of temperature and humidity and hence it is crucial to record the behavior of materials after aging. In this study, after aging at high temperatures, the tensile strength of both treated and untreated composites decreased, but the decrease was most prominent in fire retardant–treated composites. This was attributed to the degradation of the flame retardants when exposed to high temperature and humidity. This further indicated that the selection of flame retardants is an important criteria, especially for biocomposites to be used in advanced industrial applications.

The commonly used conventional FRs in the automotive sector have been described in Table 2.1.

Automotive components	Polymer	Flame retardants (FRs)
Printed circuit board	Epoxy/Phenolics	Brominated FRs; Sulfonates
Housing and dashboard	HIPS/ABS/PC-ABS alloys/ PP	Brominated FRs
Wire and cables	PP copolymers/EPR	Brominated FRs
Battery casing	РР	Brominated FRs
Textile for seats	Latex backcoating	Brominated and phosphorus- based additives
Seats	Flexible PUF	Phosphorus-based additives
Connectors and underhood parts	Polyamides	Brominated FRs
Acoustic insulation	XPE foam	Brominated and mineral- based FRs
Thermal insulation	Rigid PUF/XPE foam/ Plasticized PVC	Phosphorus-based additives
Truck and boat covers	Plasticized PVC	Phosphorus-based additives
Door partition and internal panels	PP and wood polymer composites	Mineral and phosphorus- based FRs

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 $Adapted from Fire protection for automotive and transportation. \ http://icl-ip.com/wp-content/uploads/2015/07/FR-Transportation-2012.pdf.$ 

In a recent study, researchers developed bio-based FR panels for trucks and buses from Solaris and Fiat. The biopolymers used were PHB (polyhydroxybutyrate) and PBS (polybutylene succinate), which were obtained from byproducts from the cellulose production. Sandwich panels were manufactured from natural fiber–reinforced PHB and PBS and a cork core and had potential to be used in trains and vans [30].

## 2.3.2 Use of green flame retardants

Global chemical regulations are on the increase and these changes impact a broad range of materials, including FRs. FR–based regulations are important to ensure fire safety in areas like electronics, automobiles, and furnishings. Currently some of the conventional FRs pose environmental/ecotoxicological threats, whether it is during manufacture, application, or combustion, or at end-of-life disposal, and are subsequently under the threat of being banned.

The emergence of stringent legislative measures (like REACH) and the desire for sustainability are resulting in a focus on the need for characterizing environmental and human health impacts of all chemicals. In response to the regulatory challenges, the research community has focused attention on the development of flame retardants that are safe, effective, in compliance with regulations, and are sustainable.

Some of the techniques used for imparting flame resistance by green flame retardants have been used for textile fabrics. The use of deoxyribonucleic acid (DNA) and chitosan as flame retardants through the layer-by-layer technique for cotton fabrics was investigated by Carosio et al. [31]. The authors observed a reduction of HRR by 40% during cone calorimetry tests and self-extinguishing characteristics during horizontal flammability tests. This was attributed to the fact that DNA contains a precursor of phosphoric polyphosphoric acid, a polyhydric char source and an ammonia releasing base. The combination with chitosan promoted the formation of char which reduced the flammability of the system.

Another study elaborated on the novel use of chitosan-based FR (urea salt of chitosan phosphate [UPCS]) on the flammability properties of polyvinyl alcohol (PVA). The authors observed a reduction of 42% in PHRRs upon the addition of 20% FR. This was attributed to the fact that UPCS accelerated the dehydration action and the formation of char at low temperatures [32].

In an interesting study, the flame retardance behavior of alginate fibers was studied by Tian et al. [33]. The authors reported the LOI results which showed that all zinc alginate fibers are intrinsically flame retardant, with LOI values of over 27.0, as compared with about 24.5 for alginic acid fiber. The HRR and total heat release values of zinc alginate fibers (obtained from cone calorimeter) were significantly less than those of alginic acid fiber, and decreased with increasing zinc ion content.

The use of lignin as a flame retardant for polybutylene succinate (PBS) composites was investigated by Ferry et al. [34]. Lignin possesses intrinsic properties of high thermal stability, crosslinked chemical structure, and char forming ability indicating that it can be used as a FR additive. In this study, two types of lignin (alkali and organosolv) were used and the grafting of phosphorus on lignin was also examined. It was observed that incorporation of 20 weight% of alkali lignin reduced peak HRR by 50% (Fig. 2.6) and improved charring in the PBS system. The authors conclude that modified lignin can be used as an efficient bio-based FR.

Most of the bio-based FRs developed are either expensive or affect the mechanical properties of the final product. To overcome this, in an H2020 ongoing project on bio-based FRs for consumer electronics and automotive applications, researchers are developing green FRs from a combination of a recently developed lignosulphonate flame retardant (LS-FR) and commercial ammonium polyphosphate (APP). LS-FR which is obtained as a byproduct of pulp industry has been found to be an effective and bio-based FR which com-



**FIGURE 2.6** HRR versus time curves from cone calorimeter test at  $35 \text{ kW/m}^2$  for the various PBS-alkali lignin blends.

plies with fire resistant standards for the consumer electronics and automobile sectors, while maintaining adequate mechanical properties.

## 2.4 CONCLUSIONS

This chapter deals with studies on imparting flame retardance in biopolymers and biocomposites. The introduction section gives an overview of biocomposites and stringent flammability requirements that needs to be met for biocomposites to be used in automotive and aerospace applications. The different flammability techniques and advantages and disadvantages of certain techniques have been outlined. Case studies based on flammability studies in fibers, biopolymers, and biocomposites and the use of green FRs have been presented. As seen from the case studies, the methods of imparting fire resistance has been by treating the reinforcement (natural fibers) with suitable FRs, modifying the matrix polymers by use of FR additives during compounding, and use of FR coatings. It can be inferred that FR characteristics is a prerequisite especially in advanced industrial applications and hence novel strategies for development of environmentally friendly FRs needs to be a focus area for researchers.

## REFERENCES

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