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Synthesis of A Novel Flame-retardant Hydrogel for Skin Protection Using Xanthan Gum and Resorcinol Bis(diphenyl phosphate)-coated Starch

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Firefighters continually endanger their lives in order to rescue others. This can leave them with severe burns; tens of thousands of firefighters suffered from burn-related injuries worldwide each year. Existing flame retardant fabrics cannot provide protection for the faces while superabsorbent hydrogel cannot withstand prolonged exposure to flame. Developing an anti-burn hydrogel using intumescent flame retardants would greatly reduce these risks. This research presents the synthesis of said hydrogel using biodegradable, non-toxic materials: xanthan gum (XG) and resorcinol bis(diphenyl phosphate) (RDP)-coated starch. Xanthan gum was added with RDP and RDP-coated starch and then cross-linked to produce a hydrogel. When exposed to fire, the hydrogel rapidly forms a surface char layer to insulate the heat source and to retain the ample bound water which possesses a large specific heat and enthalpy of evaporation. Flammability assessments were conducted on sheepskins and chicken skins and cumulative heat per unit area was plotted along the Stoll Curve. Results proved that 2.5% RDP-xanthan gum + 10% RDP-Starch had the optimal flame retardancy, demonstrating a prolonged skin-protection time of 45s and thus outperforming the commercial anti-burn superabsorbent hydrogel by 114%. The synthesized samples were then characterized with Thermalgravimetric analysis (TGA), viscometry, Fourier transform infrared spectroscopy (FTIR), and goniometry. The feasibility of the gel's application is supported by its thermal stability, flame retardancy, shear-thinning and hydrophilic properties, moderate pH, and non-toxicity, all of which indicated that the anti-burn gel is a viable candidate as a protective measure for not just firefighters but also for commercial use around the globe. The novel concept of employing the principle of intumescent flame retardants for anti-burn hydrogel offers new solutions to the research development in the field of flame-retardant materials.

Keywords: hydrogel, intumescent flame retardants, starch, xanthan gum, resorcinol bis(diphenyl) phosphate, skin protection

A. Introduction.

Firefighters are at a high stake of being burned when trying to save the lives of civilians; in 2017, alone, 2,835 U.S. firefighters suffered from burn-related injuries (1). Existing measures including flame-retardant fabrics are not capable of protecting faces or necks of firefighters, and there is currently no report on flame-retardant hydrogel for skin protection, which, if successfully developed, would be of great help to protect

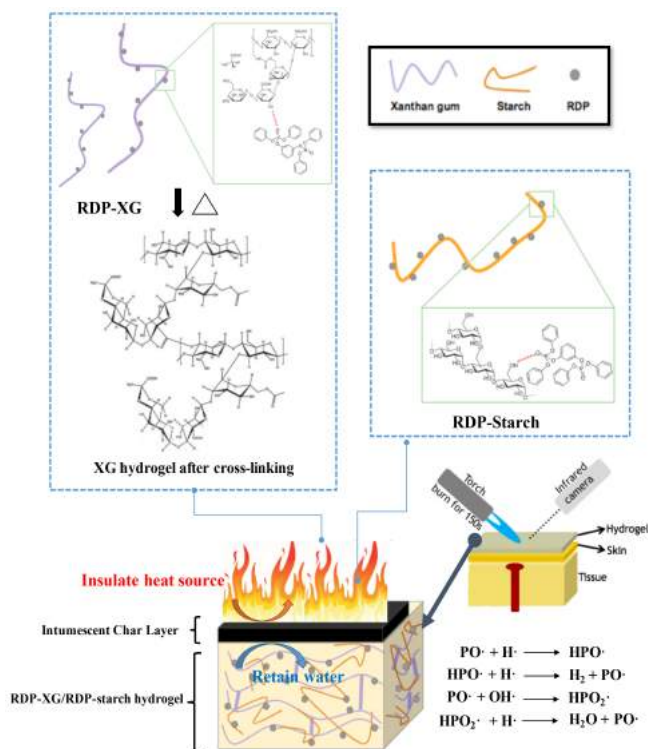


Fig. 1. Graphical Abstract

firefighters from potential burns. However, fire retardants for skin are required to be non-toxic and non-irritating. Commercial flame retardants (FRs), which mainly incorporate halogenated additives or antimony oxides (2), cannot be used due to their toxicity and potential to cause skin irritation. In recent years, intumescent phosphates have become the research focus for non-halogenated FRs due to their lower toxicity. A typical intumescent fire-retardant (IFR) system includes a carbonizing agent and an acid source to catalyze char formation (3). Since IFR systems pose significantly less threat to both humans as well as to the environment (4), they would be much more desirable for fire retardants for skin protection.

In recent years, many efforts have been made to further eliminate toxic chemicals in IFR systems. Several alternatives have been used, one of which being starch, a carbohydrate



that consists of a large number of adjoining glucose units via glycosidic bonding (5). Having a semi-crystalline structure in its natural forms, starch is inexpensive, environmentally-friendly, biodegradable, and can be found in various crops such as corn, wheat, rice, etc (6). Owing to its thermal stability and charring capabilities (7), starch has been successfully incorporated into fire retardants to enhance their performance (8–10).

Resorcinol bis (diphenyl phosphate) (RDP), a biodegradable phosphate that is found to be effective FR in readily charrable polymers in previous research (11, 12), is also being used in place of other toxic phosphates. It suppresses flames by acting as an acid precursor and acid precipitate to promote surface charring through esterification and dehydration (13). Yet, RDP is in liquid form and has high mobility, making it a challenge to reduce its bubbling under high temperatures. To address this problem, Pack et al. found that RDP is able to be attached to surfaces with hydroxyl groups, such as MMT-clay, starch, cellulose, etc (14). Using this mechanism, Guo et al. immobilized RDP by coating it to cellulose to produce a composite with both excellent mechanical properties and flame retardancy (15).

Apart from the requirement for nontoxicity, fire retardants for skin also need to be adhesive to human skin in order to function effectively. Therefore, hydrogels, instead of dry powder, would be the preferred form of fire retardants for skin due to their high affinity for skin. The formation of hydrogel requires the participation of cross-linking agents. Discovered in the 1950s, xanthan gum (XG) is a natural biopolymer that consists of a repeated unit formed by two mannose units, two glucose units, and a glucuronic unit (16). Due to its nontoxicity, high thermal stability, low flammability, and pseudoplastic rheological properties, xanthan gum has a wide range of applications in industries such as suspension stabilizer, thickening agent, and controlled drug delivery purposes, etc. (17) Previous research showed that mixing starch into xanthan gum could improve stability, viscosity, and mechanical performances of the gel (18, 19). Leone et al. prepared xanthan gum mixed hydrogel that demonstrated superior heat capacity, making it a good candidate for the cross-linking agent of flame-retardant hydrogels (20).

Herein, this work aims to decrease the risk of burned-related injuries of firefighters worldwide by presenting a novel concept for preparing skin-protection hydrogel against fire employing the principle of intumescent flame retardants; to provide an uncomplicated method to synthesize such hydrogel using an all biodegradable material combination of starch, xanthan gum, and RDP; to validate the feasibility of the synthesized hydrogel's application by investigating its thermal stability, spreadability, toxicity, and adhesiveness; and to discuss the mechanism of origin of the hydrogel's flame retardancy.

B. Materials and Methods.

Materials. Resorcinol bis(diphenyl phosphate) (RDP) used in this research was purchased from ICL (Israel Chemicals Ltd.) Industrial Products and the brand name is Fyrolflex

RDP®. Xanthan gum was obtained from Judee's Gluten-Free (Ohio, USA). Starch was purchased from local supermarkets. All Deionized (DI) water used was generated from a Milli-Q® Water Purification System.

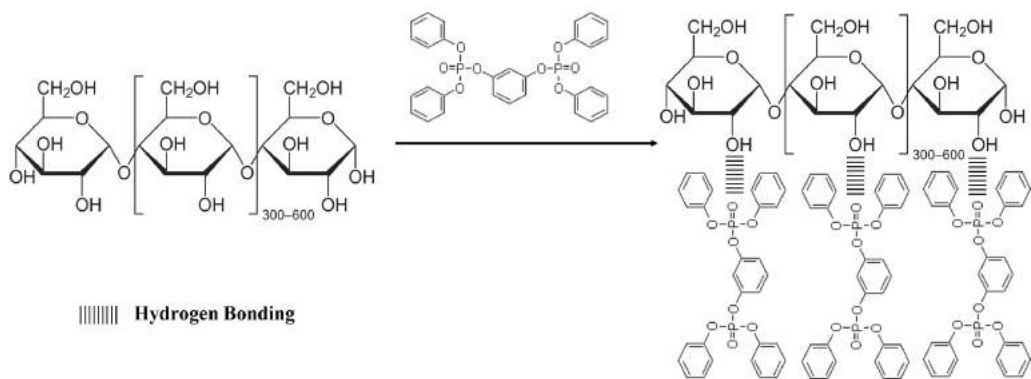
Table 1
Weight Percentage of Composition of Hydrogels

No.	Sample	XG	RDP-XG	RDP-Starch
1	1XG	1.0	0	0
2	1XG10RDP-starch	1.0	0	10.0
3	2XG	2.0	0	0
4	2XG10RDP-starch	2.0	0	10.0
5	2.5XG	2.5	0	0
6	2.5XG10RDP-starch	2.5	0	10.0
7	1RDP-XG	0	1.0	0
8	1RDP-XG10RDP-starch	0	1.0	10.0
9	2RDP-XG	0	2.0	0
10	2RDP-XG10RDP-starch	0	2.0	10.0
11	2.5RDP-XG	0	2.5	0
12	2.5RDP-XG10RDP-starch	0	2.5	10.0

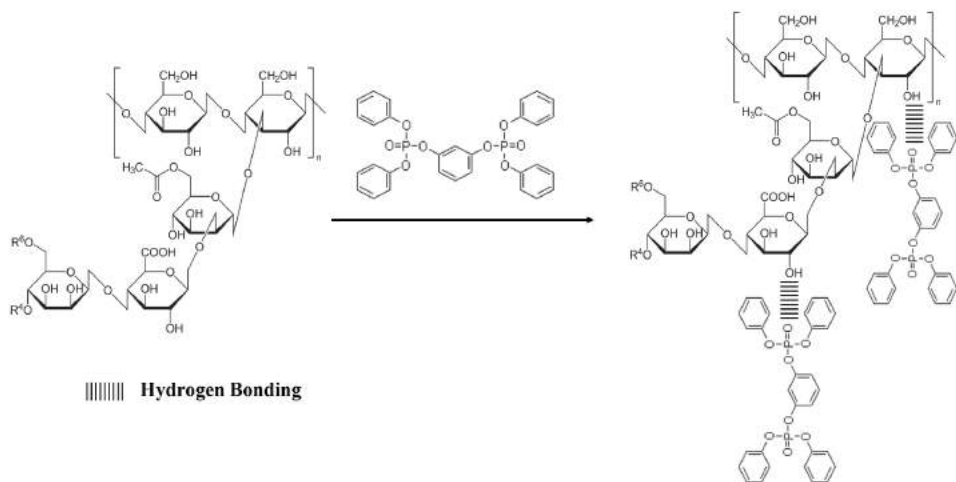
Preparation of RDP-coated starch and RDP-coated xanthan gum. In order to obtain RDP-coated starch, RDP and starch were weighed using a Mettler Toledo XPE204 analytical balance in a weight ratio of 3:7 and poured into a 200ml beaker and manually stirred with a metal spatula for 10 minutes. The beaker was kept under 40 °C for 10 minutes in a Hotpack vacuum oven. The mixed powder was then centrifuged three times using an ARE-250 THINKY centrifuge. The centrifuge was set at a speed of 750 rpm for 5 minutes and defoaming at 2000 rpm for 10 seconds. The product was kept in the Hotpack vacuum oven at 60 °C for 24 hours to remove all remaining moisture. The RDP-coated xanthan gum was prepared by first mixing RDP and xanthan gum in a weight ratio of 1:3 and then processed using the same aforementioned procedures.

Hydrogel preparation. Powders obtained from the previous section were mixed with DI water in 50 mL polypropylene centrifuge tubes to form xanthan gum (XG) based hydrogels. The samples underwent 30 seconds of shaking using a Scientific Industries Vortex-Genie 2 set at Level 10 and were then put into a VWR Incubating Waver for 10 hours to ensure thorough mixing. The temperature of the waver was set at 45 °C and the level of tilting was set at 12. Samples with different compositions of additives were listed out in the table below. Samples with higher weight percentages (3 wt.%, 3.5 wt.%, 4 wt.%) of xanthan gum were also prepared, but they failed to form uniform suspensions or hydrogels.)

Flammability assessments. A Bernzomatic Fat Boy blowtorch fueled with propane gas was used to test the flame retardant abilities of the hydrogel samples. The tests were carried out on a self-assembled stand (as illustrated in Fig.3 and consists of two parts. In part one, a piece of sheepskin



Scheme 1



Scheme 2

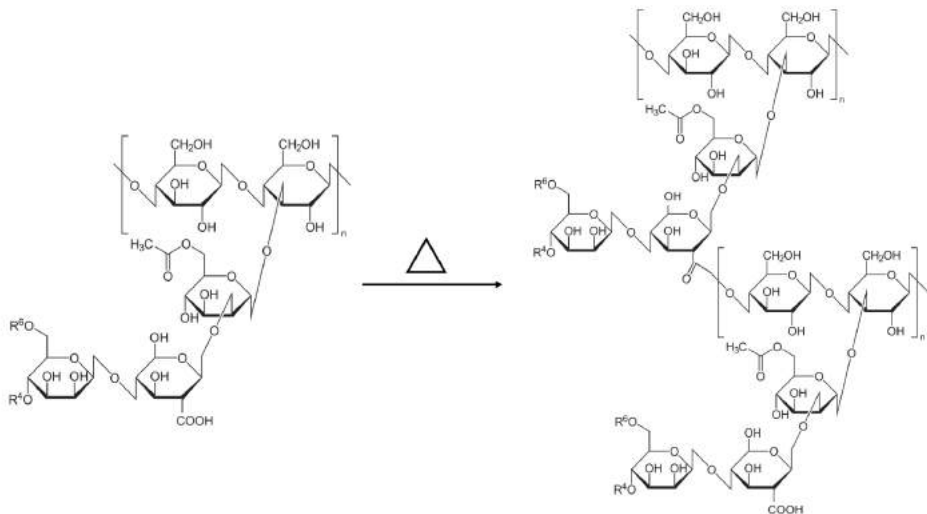


Fig. 2. Schemes for preparation of materials. Scheme 1) Formation of hydrogen bonding between starch and RDP. Scheme 2) Formation of hydrogen bonding between xanthan gum and RDP. Scheme 3) Cross-linking between xanthan gum molecules.



was cut into the size of 2cm*2cm to cover the bottom of a 2cm*2cm*0.5cm aluminum pan; 3g of hydrogel was transferred into the pan to completely immerse the skin. The pan was mounted on the stand using three spring clamps to fix positions; the distance between the surface of the samples and the tip of the blowtorch was 5cm. In part two, the sheepskin and aluminum pans were replaced with chicken skin, which was evenly cut into pieces of 3cm*3cm*1cm and applied with 3g of hydrogel. For both sections, all samples were burned with the same flame intensity for 150s and temperatures under the aluminum pan or chicken skin were recorded using an Omega thermocouple. Temperature-time curves were plotted by averaging the results from three assessments for the same sample.

Cumulative heat per unit area has also been plotted along with Stoll Criterion to determine the approximate exposure time, t_e , before receiving second-degree burn injury (21), which refers to the time length for blistering and unbearable pain to occur (22).

$$\text{Stoll curve}(J/cm^2) = 5.0204 \times t_e^{0.2901} \quad (1)$$

The cumulative heat per unit area (Q) is calculated with the following formula.

$$Q(J/cm^2) = \frac{m \times c \times \Delta T}{A} \quad (2)$$

where m is the mass of the chicken sample (g); c is the specific heat of the chicken sample [J/(g·°C)]; ΔT is the change in temperature between the temperature of the moment and the initial temperature (°C); and A is the area of the chicken sample (cm²) (23). The data of the specific heat of the chicken sample was obtained to be 1.47kJ/(g·°C).

Characterizations. Thermogravimetric analysis (TGA) was performed on a TGA/SDTA851e (Mettler Toledo, USA) to study the thermal stability of the xanthan gum-based hydrogels. About 15 to 20 mg of sample was transferred to a ceramic alumina crucible and heated from 35 °C to 750 °C at a rate of 10 °C/minute under 20 mm/min nitrogen flow. The rheological performances of the hydrogels were measured using a Bohlin Gemini HR Nano rheometer (Malvern Instruments, UK). The steady shear rate measurements were carried out in the shear rate range from 0.001 to 10 s⁻¹ and the sweep time was 100s. All measurements were conducted at 37 °C to stimulate human skin temperature. The contact angle measurements were performed with a CAM 200 Optical Contact Angle Meter (KSV Instruments, Finland). During the analysis process, a precise amount of 5 μ L of hydrogel was transferred onto a piece of sheepskin controlled by an Eppendorf™ Research pipette (Eppendorf, Germany) and the contact angles were calculated using the built-in software. Multiple droplets in each sample were measured and the results were obtained by taking the average and standard deviation of population of each trial. A Thermo Scientific Nicolet 6700 (Thermo Fisher Scientific, USA) Fourier-transform Infrared Spectroscopy was utilized to characterize pure XG, pure RDP, pure starch, 30 wt.%RDP-coated starch, and 33.3

wt.%RDP-coated XG. A calibration for the background noise was done before the actual tests. Each spectrum was an average result of thirty-two scans at a resolution and all tests are done at room temperature. The pH measurements were performed on all hydrogel samples using pH Indicator Strips (range 0-14, VWR International, USA).

C. Results and discussion.

Flammability assessments on sheepskin.

Flammability assessments on sheepskin were conducted on all the 12 XG-based hydrogel samples, the temperature of these samples at different times were recorded, and their respective temperature-versus-time graphs were depicted in Fig.4.(a) (b) (c) (d) (e). Videos of the burning tests are provided in supporting information. From the temperature curve and experimental videos, three conclusions can be generalized as follows:

- 1) Samples with higher concentrations of XG generally had better performances than those with lower concentrations. As illustrated by Fig.4, the sample with 1% RDP-XG + 10% RDP-starch had the highest final temperature compared to its counterparts with higher XG concentrations, reaching over 100 °C after 140 seconds. Its rate of temperature increase also remained stable for the shortest period of time and significantly increased at 105 seconds, at which time the flame burned out all the gel on the aluminum tin. On the contrary, the sample with 2.5% RDP-XG + 10% RDP-starch had smaller fluctuations in temperature, a longer period of stable temperature increase (25 seconds longer), and a lower final temperature (approximately 20°C lower) (Fig.4. (e)).
- 2) Given that the concentrations were the same, samples coated with RDP-starch generally performed better than ones that did not. Notably, according to Fig.4. (a), the 1%, 2%, and 2.5% RDP-XG + RDP-starch hydrogels outperformed all of its counterparts with the same concentrations in terms of final temperature and temperature stability.
- 3) The incorporation of RDP alone did not improve flame retardancy of XG gels. Increased fire retardancy can only be observed when RDP-starch is present in the gel. Fig.4 shows that, across all concentrations, RDP-XG gels had higher temperatures than pure XG gels in the entire heating process. The highest final temperature in the burning test, 213 °C, was obtained by 1% RDP-XG. Among all 12 hydrogel samples, the one with 2.5% RDP-XG + 10% RDP-Starch exhibited the best flame retardancy. It stayed below 48 °C for 50 seconds, the minimum temperature at which a first-degree burn could occur on human skin, given prolonged exposure (24), and below 55 °C for over 110 seconds, the threshold temperature for a second-degree burn to occur. Shown in Fig.5 are images of hydrogel samples at 0s, 75s, and 150s during burning and the recovered sheepskin remains after burning. When pure XG gels and RDP-XG gels were being heated, there was bubbling on the surfaces of the gels, and only small amounts of char formed and clustered at the center. Samples coated with RDP-starch exhibited less bubbling and formed thicker, more uniform char layers. Except for the 1% wt XG-based gels, all the RDP-starch coated hydrogels had sheepskin remained at

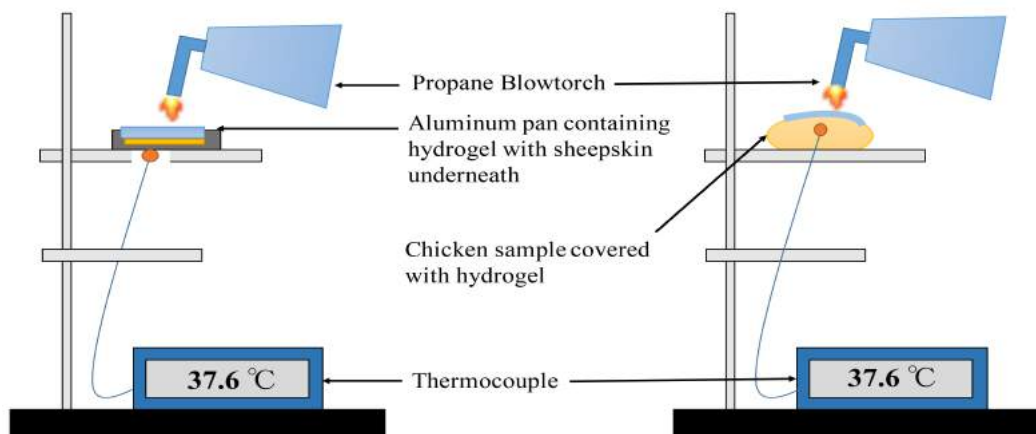


Fig. 3. Illustration of the set-up for burn tests.

the bottom of the aluminum tin. 2.5% wt. pure XG gels and 2.5% wt. RDP-XG gels also had skin remains after burning.

Flammability assessments on chicken skin. Similar assessments are also conducted on evenly cut chicken skin using the best performing samples in section one of the flammability assessments. As shown in Fig.4 and Fig.7, the chicken skin not covered with hydrogel (control) was heavily burned and the final temperature was 83°C. For the 1 wt.% XG sample, the general temperature throughout the burn test showed a significant decrease compared to the control sample, which may be attributed to the slight charring of xanthan gum and the endothermic evaporation of water. For the 2.5XG/10RDP-starch, 2RDP-XG/10RDP-starch, and 2.5RDP-XG/10RDP-starch samples, uniform char layers over the samples' surface were observed. Such char layers' formation is central to intumescent flame retardants as the char limits the spread of flame and retains the hydrogel (25). When comparing these three hydrogels, it can be identified that 2.5RDP-XG/10RDP-starch remained the best performing sample with the lowest temperature over 150s, followed by 2RDP-XG/10RDP-starch and then 2.5XG/10RDP-starch. All three samples' final temperatures were around 55°C, which is consistent with the first part of the flammability assessment. Results from a commercial flame retardant gel were also included in this experiment. It can be seen from Fig.4(f) that the commercial gel did successfully decrease the overall temperature compared to the control and the 1XG hydrogel, which can be ascribed to its superabsorbent nature (26). Yet, due to the lack of charring agent, no char layer was formed, which resulted in the loss of bound water and a rise in temperature after around 80s and thus led to a higher final temperature at 65°C than the XG-based hydrogels. 2.5RDP-XG/10RDP-starch outperformed the control by 33% and the commercial product by 21% in regard to final temperatures in the flammability assessments on chicken skin.

From the cumulative heat per unit area data plotted against time in seconds along the Stoll curve, as shown in Fig.6, it can be seen that curve of the 2.5RDP-XG/10RDP-starch sample crosses the Stoll curve at 45s, while commercial product crosses at 21s and the control, bare chicken skin, crosses at

2s. As the time of the intersection with the Stoll curve is the exposure time before receiving second-degree burn injury, 2.5RDP-XG/10RDP-starch demonstrated a prolonged skin-protection time of 45s, outperforming the commercial anti-burn superabsorbent hydrogel by 114%.

Thermal stability tests. The thermal stabilities of pure XG, RDP-Starch coated XG, RDP-XG, and RDP-Starch coated RDP-XG were investigated by thermogravimetric analysis (TGA). Four main thermogravimetric characteristics in a single stage of thermal degradation are shown in Table 2, i.e. T_{onset} , the temperature at which an observable thermal decomposition starts (1% weight loss); T_{max} , the temperature at which the rate of thermal degradation reaches its local maximum; T_{endset} , the point at which a stage of thermal degradation is complete; W%, the weight percentage loss during each stage. (27) As presented in Table 2, the cross-linking between RDP and XG did not affect the thermal stability of the hydrogels, as both XG and RDP-XG gels only displayed a single-stage weight loss from 90 °C to 110 °C, which is attributed to the loss of both loose and bound water (28). Compared to pure XG samples, the RDP-coated sample had a slightly higher T_{max} and T_{endset} . After the first stage, XG and RDP-XG samples had residues of approximately 3% and started to decompose slowly as the temperature increased. In the 260 °C– 350 °C range, RDP-coated XG sample displayed a higher rate of decomposition, as shown in Fig.8 below. Previous studies have shown that this was caused by the thermal decomposition of RDP. (29) The presence of starch, however, significantly enhanced the thermal stability of XG-based gels. After the evaporation of bound water, samples coated with RDP-starch had higher residues of over 10% and further underwent a second-stage thermal decomposition from 250 °C to 360 °C, in which both RDP-starch coated RDP-XG and RDP-starch coated XG samples displayed 5%–9% weight losses. Mass loss in this stage is caused by the degradation of starch, as pure starch also degrades at this temperature (Fig.8.a). In general, the RDP-XG/RDP-starch demonstrated a relatively high thermal stability, which would guarantee the absence of toxic volatile at a low temperature, adding to the application value of the gel.

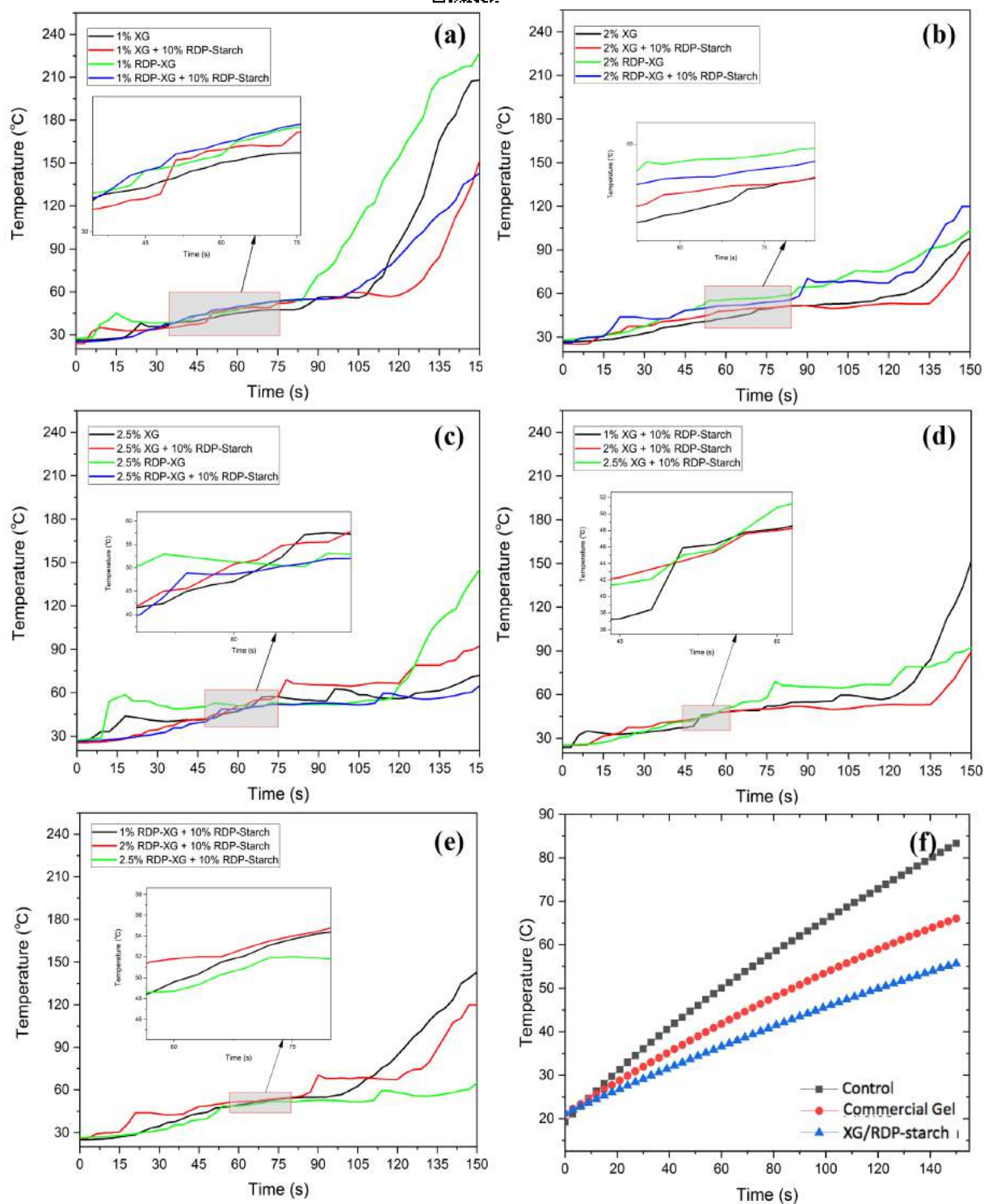


Fig. 4. Temperature-versus-time graphs of all hydrogel samples on sheepskin and chicken skin. a) Samples with 1% XG on sheepskin. **b)** Samples with 2% XG on sheepskin. **c)** Samples with 2.5% wt. XG on sheepskin. **d)** Samples with XG + RDP-Starch on sheepskin. **e)** Samples with RDP-XG + RDP-Starch on sheepskin. **f)** Samples on chicken skin

Rheology analysis. The viscosity is plotted as a function of shear rate to understand the rheological properties of the hydrogels. As these hydrogels are required to be applicable to the skin, thixotropic, the property of viscous gels to flow when stressed, is a crucial parameter for the evaluation of their application practicality. According to Fig.9, viscos-

ity decreased with the increase of shear rate, indicating the shear-thinning behavior of all samples. Since shear-thinning is a special case of thixotropic behavior, which takes zero-time to return to the liquid's initial stage (30), the feasibility of being applied to the skin is therefore validated. Moreover, it can be noted from Fig.9. (a)(b)(c) that the higher the weight



Fig. 5. Hydrogels at 0s, 75s, and 150s of flammability assessments and the recovered sheepskin remains.

percentage of XG or RDP-XG, the more viscous the hydrogel. Also, under the condition that the XG concentrations were the same, samples with RDP-starch were always the ones with the highest viscosity. The addition of RDP-starch increased the viscosity of both XG or RDP-XG hydrogels, which is in line with the finding of Abduloma et al. that the overall viscosity of xanthan-starch mixture would be greatly enhanced because xanthan promotes the association between the mixture's granules (18). In addition, the coating of RDP, if reaching a certain amount, would decrease the viscosity (as shown by Fig.9. (b)(c)), and this may be attributed to the liquid nature of RDP. Thus, referring to Fig.9. (d) and under the

knowledge that viscous gels adhere better to human skin (31), it is determined that 2.5%XG+10%RDP-starch has the optimal rheological performances in terms of viscosity, followed by 2.5%RDP-XG+10%RDP-starch.

FTIR Spectra. Fourier-transform Infrared Spectroscopy was employed to investigate the formation of hydrogen bonding between RDP and XG/starch. As depicted in Fig.10, the spectrum of pure xanthan gum displayed a broad peak at around 3300 cm^{-1} , indicating the presence of O-H bonds in the alcohol groups of xanthan gum(32), whereas pure RDP showed no absorption in this region. In the FTIR spectrum

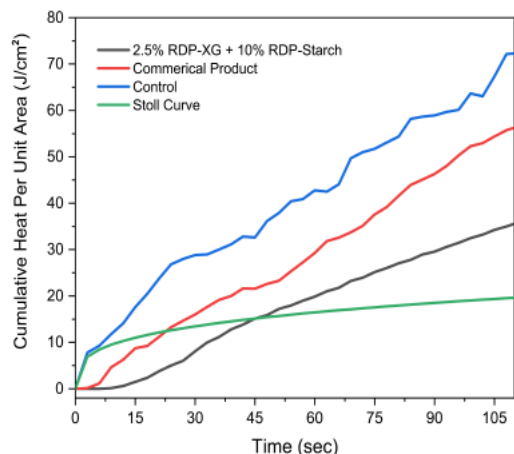


Fig. 6. Cumulative heat per unit area of against time curves of the control, commercial anti-burn superabsorbent hydrogel, and the synthesized 2.5RDP-XG/10RDP-starch plotted along the Stoll curve.

Table 2

Four main characteristics of thermalgravimetric analysis

No.	Stage	Tonset (°C)	Tmax (°C)	Tendset (°C)	Loss (%)	Residue (%)
5	1	38.9	101.4	118.1	97.4	2.61
6	1	37.0	93.7	143.0	89.6	10.4
	2	276.2	304.2	353.4	5.89	3.29
11	1	36.3	107.8	127.9	96.9	3.07
12	1	38.9	91.1	130.4	86.1	13.9
	2	276.9	293.9	370.4	8.76	3.24

of RDP-XG, the transmittance curve exhibited a similar pattern, but the peak in the same region was broadened and had a higher absorbance rate. This is an indication of an increase in bond lengths of O-H covalent bonds due to their involvement in hydrogen bonding between RDP and XG (33). Examination of RDP-Starch yielded similar results, as depicted in Fig.10. Again, the broadening and deepening of the transmittance curve between 3100 cm^{-1} and 3600 cm^{-1} strongly suggests the existence of hydrogen bonding between RDP and starch.

Goniometry. The contact angles of hydrogel samples on the sheepskin were determined through goniometry to simulate the mechanical behavior of the gels on human skin. The test data are shown in Fig.11 below and the contact angle images of the four best performing samples in the burn tests are shown in Fig.12. As hydrophilic surfaces are known to have contact angles between 10° and 90° , this result demonstrated the outstanding hydrophilicity properties (34), which may be attributed to the fact that water accounts for a significant weight percentage of the hydrogels. The average contact angles of all samples were approximately between 45° to 60° , which are comparable with the contact angles of water on human skin (between 50° and 60°)(35), proving the

spreadability and applicability of the hydrogel to the human skin.

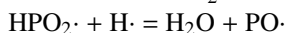
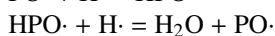
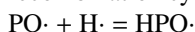
pH test. pH tests were conducted on all XG-based gels in order to test their potential to cause skin irritation. The pH values of all the hydrogel samples are shown in Table 3. All samples proved to be neutral to slightly acidic, having pH values between 5 and 7. The presence of phosphoryl groups makes RDP slightly acidic; and when added into XG gels, RDP lowers the pH value of the flame-retardant gels. Starch also slightly reduces the pH values of the gels, but the slight acidity of RDP and starch will not do harm to human skin in that natural skin surface pH is on average below 5(36).

Table 3

pH Values of the Hydrogel Samples

Sample	pH Value
1XG	7
1XG10RDP-starch	6
2XG	7
2XG10RDP-starch	6
2.5XG	7
2.5XG10RDP-starch	6
1RDP-XG	6
1RDP-XG10RDP-starch	5
2RDP-XG	6
2RDP-XG10RDP-starch	5
2.5RDP-XG	6
2.5RDP-XG10RDP-starch	6

Flame retardancy mechanisms. Based on the analysis above and previous research on the flame retardancy mechanisms of RDP, the plausible flame retardancy mechanism of RDP-coated XG+ RDP-Starch is illustrated in Fig.13. When RDP-coated XG+ RDP-Starch was exposed to the flame, the cross-linking between molecules broke, followed by the vaporization of bound water in the hydrogel. As the temperature increased, starch and XG experienced dehydration process to form ether segments (37), while at the same time RDP was heated into the gas phase, releasing PO, PO₂, HOPO, and HOPO₂ radicals (38) that participated in the catalytic radical recombination cycle to inhibit the flame:



Here, RDP served as an acid agent and promoted the charring process of XG and starch. At approximately 300°C , XG and starch underwent further dehydration to produce aromatic rings, such as phenol, benzene, and furan structures with either CH₂ or CH₂-O-CH₂ as main linkages in between (37). Over 600°C , carbonization took place as large conjugated aromatic structures were formed and were eventually turned into amorphous carbon structures. The overflow of H₂O and gaseous radicals resulting from RDP decomposition caused to char layer to possess an intumescent structure.

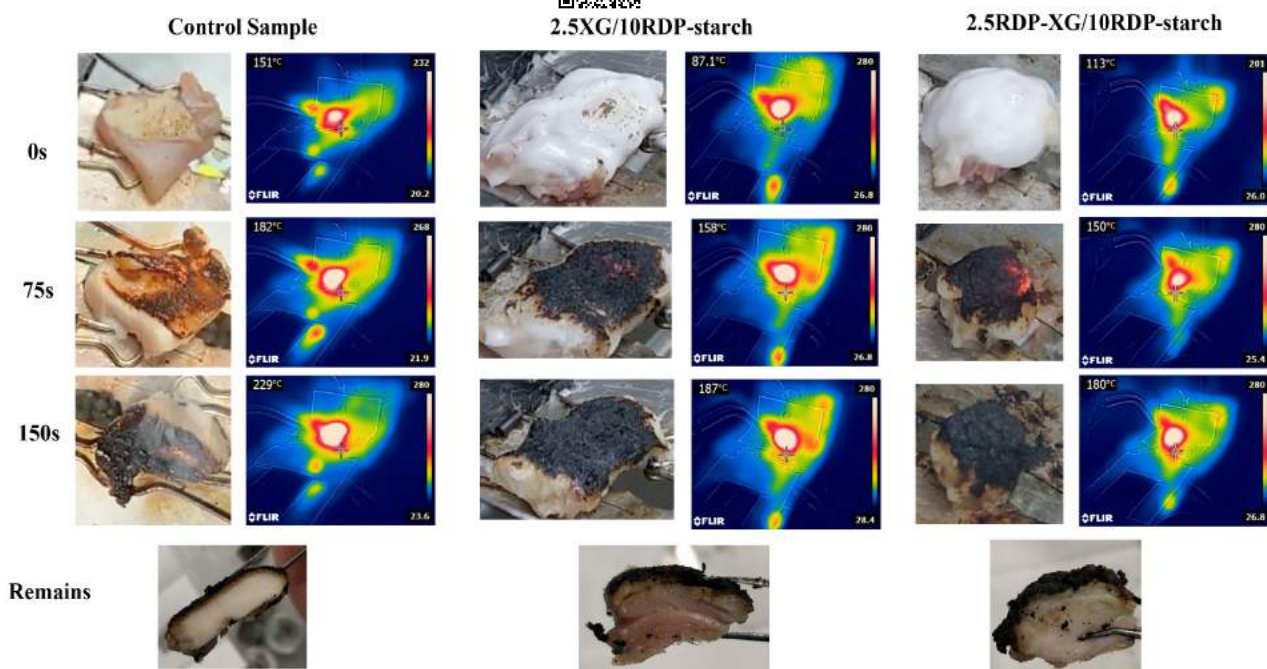


Fig. 7. Pictures and Infrared Pictures of Chicken samples at 0s, 75s, and 150s of the flammability assessment and cross sections of the samples after burning.

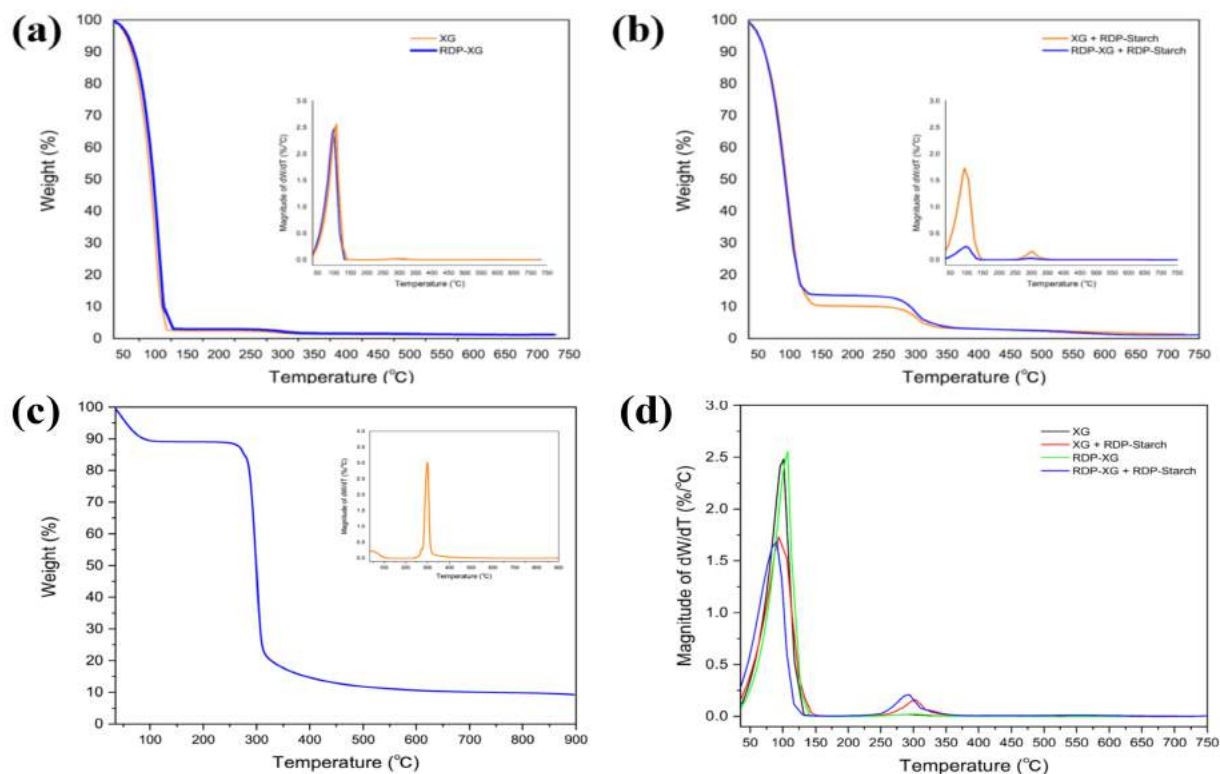


Fig. 8. Results for thermogravimetric analysis (shown in the insets are the magnitude of the original curves' first derivatives). a) TGA curves of 2.5XG gel and 2.5RDP-XG b) TGA curves for 2.5XG/10RDP-Starch and 2.5RDP-XG/10RDP-Starch c) TGA curve for pure starch. d) First derivative of TGA curves of XG-based gel samples.

Therefore, an even residue char structure was formed on the surface of the hydrogel, isolating heat radiation from the heat

source and protecting the hydrogel underneath. The presence of starch reduced the bubbling of RDP when the hydrogel

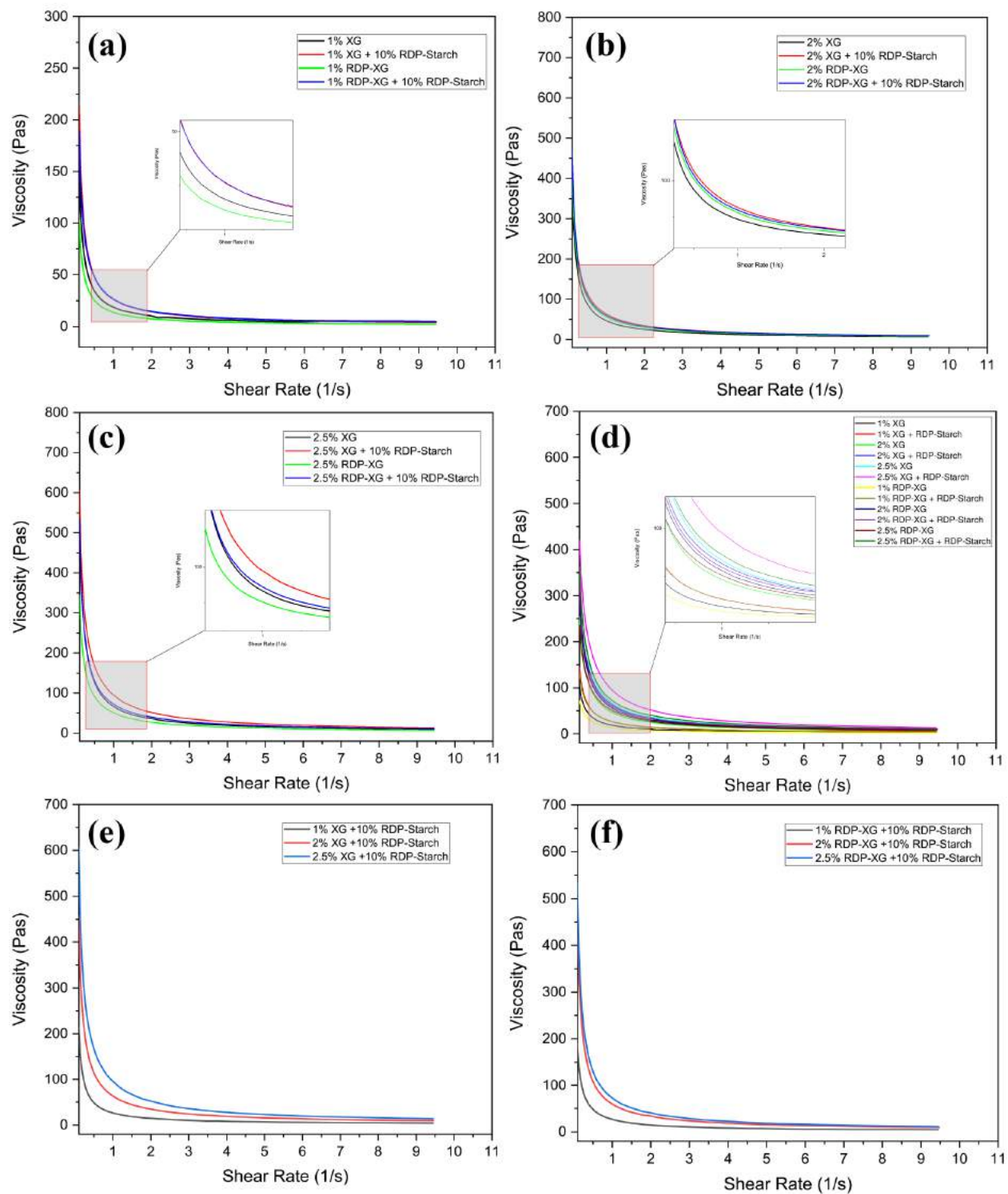


Fig. 9. Rheological test results of the hydrogel samples. a) samples with 1 wt.% XG or RDP-XG. b) samples with 2 wt.% XG or RDP-XG. c) samples with 2.5 wt.% XG or RDP-XG d) all samples e) samples with XG + 10% RDP-starch f) samples with RDP-XG + 10% RDP-starch

was exposed to flame and increased its overall flame retardancy, proving that starch enhanced the trapping effect on the gases formed during heating.

Discussion of toxicity. Although RDP has been listed as a flame retardant material with minimal toxicity by the United States Environmental Protection Agency (US EPA), there are

growing concerns regarding the safety of human exposure to this material. Recent research reported that RDP may have a low to moderate toxicity humans, high to very high aquatic toxicity (*Daphnia magna* 48-h EC₅₀ = 0.7 mg/L), moderate persistence, and high bioaccumulation potential (39, 40). However, a study in 2015 shows that when RDP is absorbed to a substrate like the MMT clay, the toxicity of the RDP

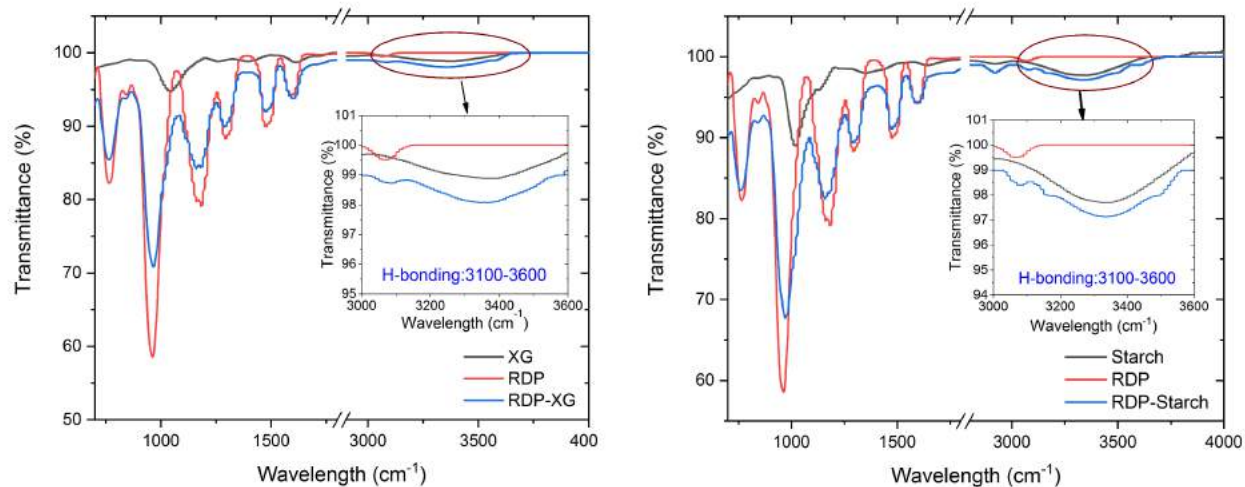


Fig. 10. FTIR spectra of hydrogel samples. a) pure xanthan gum (XG), pure RDP, RDP-coated XG. b) pure starch, pure RDP, and RDP-coated starch

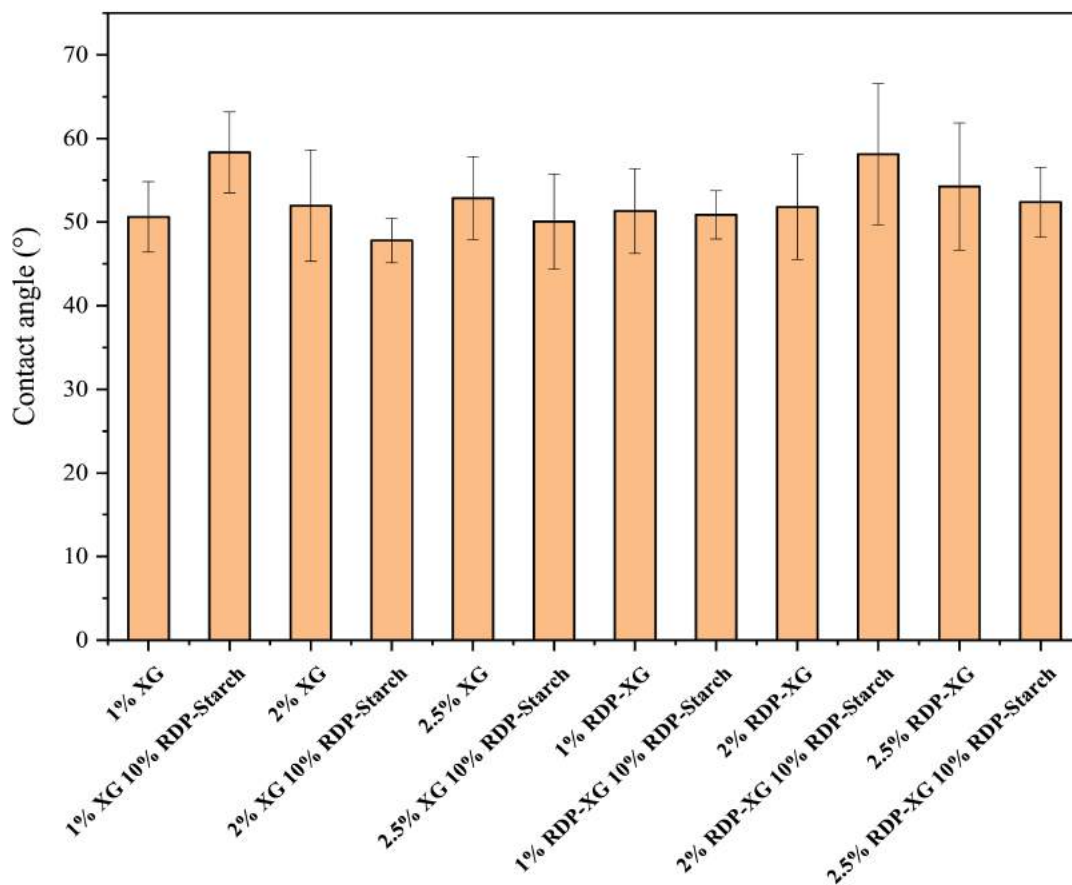


Fig. 11. Average contact angles of hydrogel samples on sheepskin.

would be greatly reduced (41). In the mentioned study, human dermal fibroblasts were placed on top of coatings of RDP/polymer/clay on a glass slip (PS is the abbreviation for polystyrene). Cells were then cultured and the doubling times were recorded and compared. As shown in Fig.14, RDP in fact promotes the proliferation of dermal fibroblasts, and the

doubling time on the coating containing RDP is the same as that of the plastic control. Therefore, it can be drawn from the results that absorbed RDP is non-toxic to human dermal fibroblasts, rendering this study's hydrogel safe to apply on human skin.

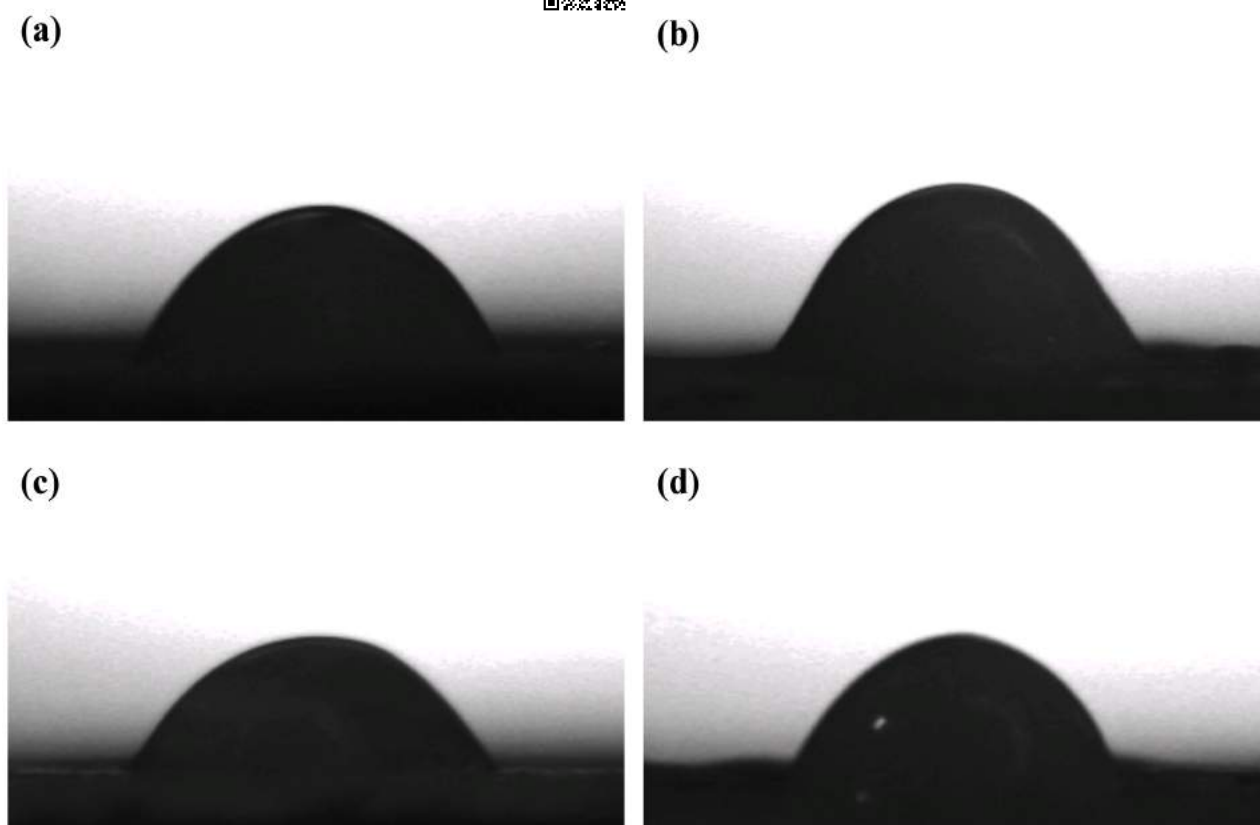


Fig. 12. Contact angles of hydrogels on sheepskin of the four best performing samples in burn tests. a) 2.5XG/10RDP-starch. b) 2.5RDP-XG/10RDP-starch. c) 2.5XG. d) 2 RDP-XG/10RDP-starch.

mechanism.png

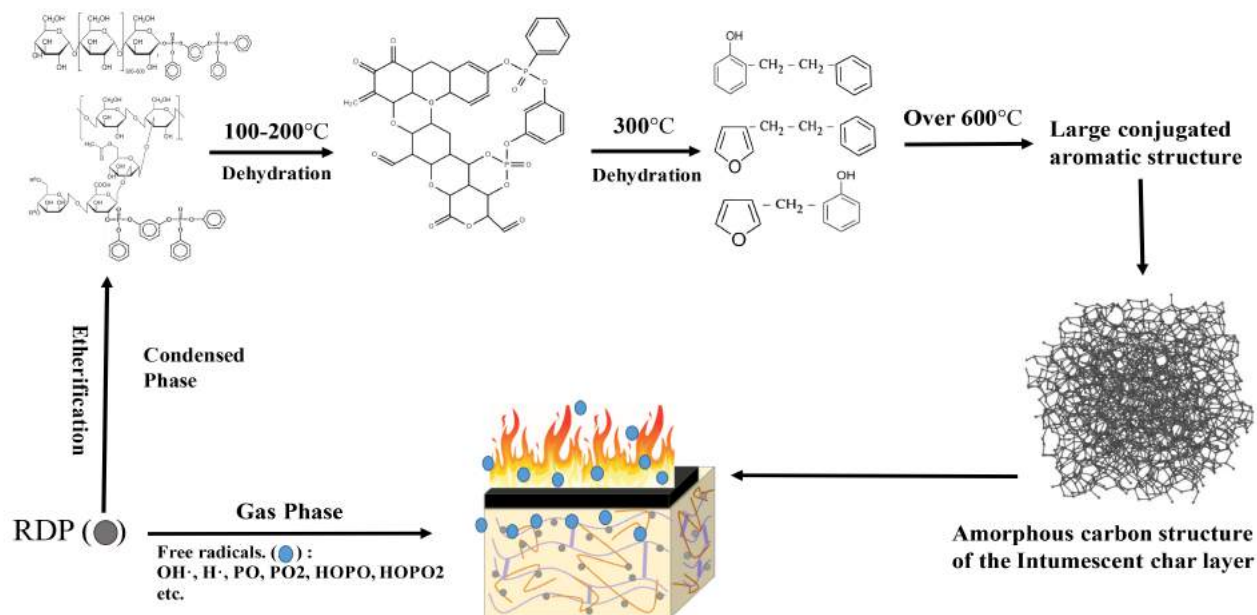


Fig. 13. Illustration of flame-retardant mechanism of RDP-XG/RDP-starch hydrogel

Future work. In order to continue to enhance the performance of the anti-burn hydrogel, further research should be concentrated on 1) optimization on the RDP to starch ratio and the amount of loading of RDP-starch in gel; 2) Gas chro-

matography-mass spectrometry (GCMS) analysis on gels to identify volatile products and burning residue; 3) analysis of the absorption of RDP to human skin and possible irritation; 4) designing a thermal protective performance test for the

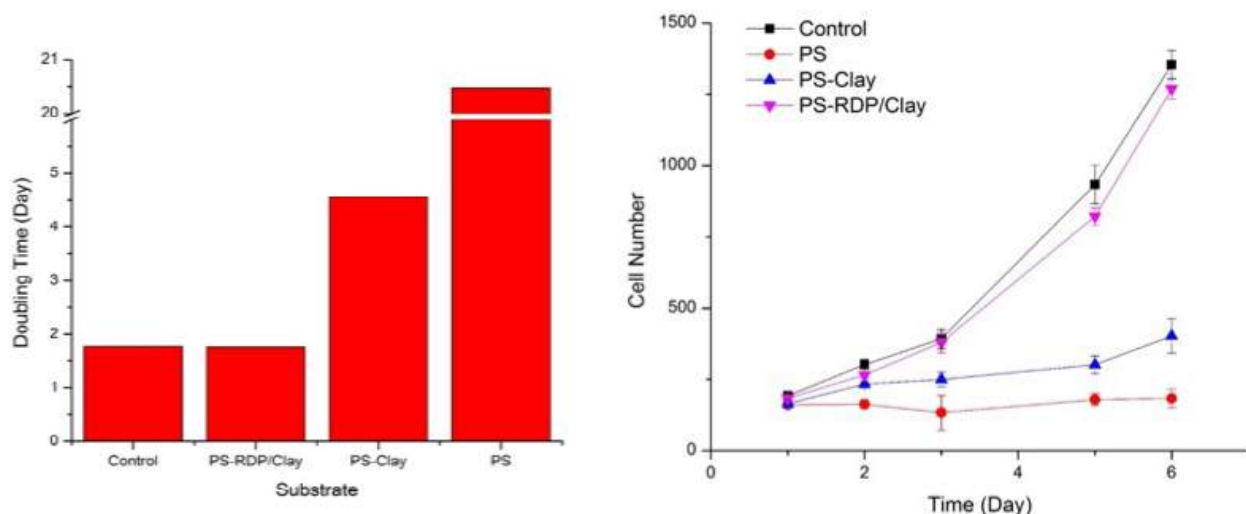


Fig. 14. Growth and doubling time of human dermal fibroblasts on different substrates

hydrogel and compare the result with Stoll curve to determine the time before causing second-degree burn; 5) analyzing the char residue with FTIR and SEM to determine the exact mechanism of the flame retardancy

D. Conclusion. In conclusion, this study has shown that a biodegradable, non-toxic, non-irritating flame retardant hydrogel for skin protection can be engineered by blending starch coated with RDP into xanthan gum-based hydrogel. The incorporation of starch increased the general thermal stability of the material, stabilized RDP by preventing its bubbling, and rendered RDP nontoxic by fixing its phenyl groups to a substrate. The formulation 2.5RDP-XG/10RDP-starch possesses the best performance in regards to hydrogel uniformity and flame retardancy. When burned for 150s, the bottom sheepskin covered with the gel stayed below 48 °C for 50s and below 55 °C for over 110s. When tested on chicken skin, the gel formed an even char layer and kept the skin well-preserved. The bottom final temperature reached only 55.9°C and the skin-protection time before receiving second-degree burn reached 45s, outperforming the commercial anti-burn hydrogel by 114%. Characterization results revealed the hydrogel's thermal stability, flame retardancy, shear-thinning and hydrophilic properties, moderate pH, and non-toxicity, all of which indicated that the anti-burn RDP-XG/RDP-starch gel was easily spreadable and safe to use as a protective measure for firefighters worldwide. The novel concept of employing the principle of intumescent flame retardants for anti-burn hydrogel offers new solutions to the research development in the field of fire safety materials science.

ACKNOWLEDGEMENTS

We would like to acknowledge the support from the Louis Morin Charitable Trust and NYS Department of Economic Development. We are sincerely grateful to our mentors, Dr.Miriam Rafailovich and Yuan Xue, for their guidance on the use of instruments and knowledge on testing methods; for the provision of the research laboratory for two months at Stony Brook University College of Engineering and Applied Sciences; and for their advice in writing the thesis and characterization analysis. We would also like to thank Dr.Jonathan Sokolov for providing us with the instruments for the flammability assessments; thank Lisa Quinto, Jalaj Mehta, and Lauren Stiefel for their helpful advice; thank all the faculty of Stony Brook Uni-

versity for giving us a fulfilling summer at the 2019 Garcia Center for Polymers at Engineered Interfaces Summer Research Program (Garcia MRSEC).

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EDUCATION

Guangzhou Tianhe Foreign Language School - High School

Sep 2014 - Jun 2019

- Grade Ranking: 3 out of 199

- SAT 1540, test date: March 9th, 2019

(Evidence-Based Reading and Writing: 740, Math: 800, Essay: Reading 7, Analysis 6, Writing 7)

- TOEFL 110, test date: April 13th, 2019 (Reading: 30, Listening: 30, Speaking: 26, Writing, 24)

- SAT Subject Tests: Math II (800), Chemistry (800), US History (800), test date: June 1st, 2019

- Advance Placement Courses: 2018: AP Macroeconomics 5, AP Microeconomics 5; 2019: AP Environmental Science 5, AP Calculus BC 5, AP Chemistry 5

RESEARCH EXPERIENCE

Stony Brook University - Garcia Summer Research Program

Jun 2019 - Present

- Conducted research in the labs of Prof. Miriam Rafailovich on flame retardant materials with the purpose of saving the lives of firefighters
- Project: Synthesis of A Novel Flame-retardant Hydrogel for Skin Protection Using Xanthan Gum and Resorcinol Bis(diphenyl phosphate)-coated Starch
- The project is well received after presenting at Israel Chemicals Ltd. and Stony Brook Fire Dept.

School of Chemistry, Sun Yat-sen University - Science Talent Program

Dec 2017 - Present

- Conducted research in the lab of Prof. Yexiang Tong on photocatalyst and electrosorption with funding supported by China Association for Science and Technology (CAST)
- Project: "Absorption and Photodegradation of methyl orange with BiOI/BiOBr/MoS₂ composites under visible light irradiation" and "Preparation and Electrosorption Desalination Performance of Peanut Shell Based Activated Carbon and Defect-rich MoS₂"
- Selected as a member of the 2018 Japan-Asia Youth Exchange program in Science (SAKURA Exchange Program in Science)
- Represented China in 2019 International Competition for Young Scientists (Unge Forskere), awarded the third prize
- Selected as the only delegate to represent China for 2019 Stockholm Junior Water Prize

School of Mathematics, Sun Yat-sen University - Science Training Camp

Jan 2018 - Jul 2018

- Group Project "Classification of ECG signals based on deep learning" (as the first author)
- Awarded the best project in Mathematics and Computer Science in the program symposium

LEADERSHIP EXPERIENCE

Student Union - President

Jan 2018 - Jul 2019

- Drafted the founding documents of the student union
- Pioneered the Tianhe District Interschool Summit, invited the presidents and council members of nine other schools to join (50+ attendees)
- Led the young union to host many of its first campus activities: student-principle forums, the "How's That" talent show, the THFLS Basketball Association, the debate competition, the Flee Market for Charity (Raised over 20kRMB), the National Day Singing Competition, etc. (each activity has 500+ attendees)

the Scary (Car Designing Startups) - CEO

Sep 2017 - Jul 2018

- Supported by Toyota and led the 6-person startups to design and produce a car model using 3-D printing
- Held a press conference with over 300 attendees and attracted media reporters
- Spoken highly of by the chief designer of Guangzhou Toyota

TW Drones - Club Founder



国际竞赛 科研科创 发表论文
关注“有方背景提升”

Oct 2016 - Jul 2018

- Founded the club with two other roommates and secured funding from the school
- Established connections with South China Agricultural University Drone Club, thus providing club members with official training at university labs
- Club members won nine gold medals in 2019 Guangdong Championships of Aerospace Model

COMMUNITY SERVICES

Nepo: Drinking Water Protection Society - Founder

Feb 2018 - Present

- started the organization with the objective of protecting drinking water sources and teaching people about its methods and importance
- investigated the water quality of the local water source, Liuxi River, and made suggestions to the municipal government for the river's better protection
- the organization researched on a cheaper way of ammonia nitrogen's removal using chlorination and published the work on a peer-reviewed journal

"The Next Stage" Music Drama Club - Member, Leading role

Sep 2016 - Jul 2018

- Staged performances under the guidance of professional British musical actors
- Performed at three charity fairs to raised money for poor children from western China

PUBLICATIONS

- Photocatalytic degradation of methyl orange by BiOI/Bi₄O₅I₂ microspheres under visible light irradiation, Inorganic Chemistry Communications, SCI, 93(2018) 65-68. (English, Third author)
- Influential factors of water environment quality and the protection measures of Liuxi River in Baiyun Section, Environment and Development, 6(2018), 6-8. (Chinese, First author)
- A new way of ammonia nitrogen removal - determination of ammonia nitrogen content in water and removal of ammonia nitrogen by chlorination method, invention and innovation (the juvenile student) 10(2018)10. (Chinese, First author)

HONORS & AWARDS

- Finalist for the Awarding Program for Future Scientists organized by CAST Sept. 2019
- 2019 Stockholm Junior Water Prize Finalist Aug. 2019
- GENIUS Olympiad Gold Medal in Science, Oswego, NY, USA Jun. 2019
- Champion of China Junior Water Prize, Nanjing, China May. 2019
- Third Prize in International Competition for Young Scientists (Unge Forskere), Copenhagen, Denmark Apr. 2019
- Champion of Novice Division of National Speech & Debate Association (China) Regional Championship, Guangzhou, China Apr. 2019
- First Prize in Guangdong Chemistry Experimental Competition, Guangzhou Jun. 2018
- Hackathon Third Place for the redesign of public spaces at Harvard and Individual Third Prize in China Thinks Big Global Final, Cambridge, MA, USA Apr. 2018
- National Top 10 of Rising Star China Chemistry Challenge of Royal Society of Chemistry, Beijing, China Mar. 2018

SKILLS, CERTIFICATIONS & OTHERS

- **Skills:** Python (Basic), OriginLab (Proficient), Photoshop (Proficient), MIT App Inventor (Advanced), 123DX (Advanced)
- **Interests:** Soccer (Class team member), Saxophone (School orchestra member), Photography (2nd Prize in Guangzhou Photography Competition for Teenagers), Birdwatching (3rd Prize in Bird-watching Competition of Guangdong)



国际竞赛 科研科创 发表论文
关注“有方背景提升”

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HIGH SCHOOL ACADEMICS

Shanghai Starriver Bilingual School (Sep 2017— present)	GPA: 4.459/4.691 (weighted) (top 5%)	AP Courses: Chemistry, Computer Science A, Calculus BC, Environmental Science (self-study), Physics 1, Physics C: Electromagnetics (self-study)
Standardized Test Scores	TOEFL: SAT:	110 (R: 29, W: 29, S: 23, L: 29) 1530 (Math: 780, EBRW: 750, Writing: 20)

COLLEGE LEVEL ACADEMICS

AP Capstone Seminar & Research (Jan 2019—present)	Ongoing: An Interdisciplinary Project Including Chemistry	<ul style="list-style-type: none">Working with a selective research team consisting of 11 top students and 4 AP teachersConducting an interdisciplinary project that involves Chemistry, Biology, and Economics
Sun Yat-sen University (Jul 2018—Aug 2018)	Chemistry Research Project: Carbamoyloxylolation of carboxamides (*See below 1)	<ul style="list-style-type: none">Analyzed 30+ molecular structures through ^1H NMRDesigned and experimented 10+ synthetic routes independentlyIntegrated experimental data and translated experimental reports into EnglishSupported analyzing the components of impuritiesResearch paper submitted to <i>Organic Letters</i>
Sun Yat-sen University (Jul 2017—Aug 2017)	Chemistry Research Project: Decarboxylation Of Lactones (*See below 2)	<ul style="list-style-type: none">Received training on experimental methods of organic synthesisConducted 10+ experiments and collected experimental data which were then directly used in the final research paperSynthesized reagents for the final reactionStudied organic chemistry textbooks for college freshmenResearch paper published in <i>Green Chemistry</i> 20.4(2018): 804-808

1: "Copper-Catalyzed C-H Carbamoyloxylolation of Aryl Carboxamides with CO_2 and Amines at Ambient Conditions"

2: Huang, Xihua, et al. "Decarboxylation of α , β -unsaturated aromatic lactones: Synthesis of E-ortho-hydroxystilbenes from 3-arylcoumarins or isoaurones." *Green Chemistry*. 20.4(2018):804-808.

HONORS & AWARDS

- National Silver Award* at Royal Society of Chemistry: [RSC3 Chemistry Contest 2018](#)
- Regional Award* at Royal Society of Chemistry: [RSC3 Chemistry Contest 2017](#)
- National Finals* at China Thinks Big (CTB) Contest 2017
- Outstanding Presentation* at China Thinks Big (CTB) Contest 2017
- Outstanding Studies Award* (top 10% in school) in 2017, 2018.



国际竞赛 科研科创 发表论文
关注“有方背景提升”

- *Innovation Award* (top 8% in school) in 2017, 2018
- *Outstanding Student Club Award* in 2018
- *Second Award* at School's Singing Contest

LONG TERM SCHOOL ACTIVITIES

Sep 2018—present

Shanghai Starriver Bilingual School (SSBS) Chemistry Club

- Taught fundamentals of organic chemistry (basic naming and reactions)
- Led 15+ members to conduct classic reactions such as producing aspirin and silver mirror reactions, etc.

Sep 2018—present

ASB Regulation Department (Student Council)

- President of regulation department; managed a committee of 20 members
- Promoted and secured the execution of school regulations

Sep 2017—present

SSBS Rubik's Cube Club

- Founder and president of the club
- Held 30+ weekly activities, designed a unique tutorial for Rubik's cubes, and taught students to solve cubes
- Brought the element of Rubik's cubes to a series of school activities, including new year show, charity fair, club fair, etc.

Sep 2017—present

SSBS Energy Club

- Responsible for setting up the stage for all the school performances
- In charge of sound and light control during performances

COMMUNITY SERVICE & EXTRACURRICULAR

Aug 2018

Harvard College AUSCR Summit for Young Leaders in China (HSYLC)

- A two-week program with three intense college-level courses and a capstone project on machine learning: GPA 3.86/4.0 (top 15%)
- Spent 10+ hours editing a promotion video
- Performed on the closing ceremony in front of over 400 people.

Oct 2017—Mar 2018

China Thinks Big (CTB) Contest

- Leader of a 4-membered team; conducted a project on shared parking
- Creatively designed a "shared parking platform" for communities in Shanghai
- Performed investigations on more than 35 commercial parking lots; conducted field surveys to over 30 local housing estates
- Proposal under consideration by Shanghai Transport Administration Commission

Spring 2018

Voluntary Online Support Education

- Taught 20+ children from a remote countryside in China (Liutai Village)
- Prepared the plan and content for the 4-week course

SKILLS

Extremely skilled in solving Rubik's cubes—personal record on World Cube Association (WCA):13.49 seconds for 3x3 single (World Rank: top 9%)

Proficient use of Adobe Premiere (PR), Aftereffects (AE), Photoshop (PS), and Indesign (ID)



Resumes of mentors

1. Miriam Rafailovich

Wikipedia Page: https://en.wikipedia.org/wiki/Miriam_Rafailovich

Miriam Rafailovich (born October 29, 1953) is a materials engineering researcher. She is the director of the Garcia Materials Research Science and Engineering Center at Stony Brook University as well as former co-director of the Chemical and Molecular Engineering program at Stony Brook University. Her publications focus mainly on nanoscale materials engineering, including nanofibers, supercritical carbon dioxide, and biodegradable polymers.

Education:

- Bachelor of Science degree, Brooklyn College, 1974
- Ph.D in nuclear physics, SUNY Stony Brook, 1980
- Languages: English, Romanian, Hebrew, Yiddish, French, and German.

Publications

- Google scholar page: <https://scholar.google.com/citations?hl=en&user=biedycAAAAJ>
- Citations: 18214; h-index: 72; i10-index: 275

Contacts

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- Tele: +1 (516) 458 9011
- Add: Department of Materials Science and Chemical Engineering, Stony Brook University, Stony Brook, NY 11794, USA

2. Yuan Xue

Education

- Bachelor of Material Science and Engineering, Nanjing University of Science and Technology, 2012
- Master of Mechanical Engineering, Stevens Institute of Technology, 2014
- Ph.D. in Material Science and Engineering, Stony Brook University, 2019

Publications

- Google scholar page: https://scholar.google.com/citations?hl=en&user=_TkBUDIAAAAJ
- Citations: 108; h-index: 6; i10-index: 5

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