

## Supplementary material

**Table S1**–Constant SASD parameters.

F <sub>CO<sub>2</sub></sub>	F <sub>Feed</sub>	T <sub>in</sub>	T <sub>SM</sub>	Th <sub>CO<sub>2</sub></sub>	T <sub>CCO<sub>2</sub></sub>	P <sub>SM</sub>	d <sub>nozzle</sub>
(mL/min)	(mL/min)	(°C)	(°C)	(°C)	(°C)	(MPa)	(μm)
25	3.5	100	80	80	-20	10	150

F<sub>CO<sub>2</sub></sub>: CO<sub>2</sub> flow rate; F<sub>Feed</sub>: liquid feed solution flow rate; T<sub>in</sub>: inlet drying gas temperature; T<sub>SM</sub>: saturation temperature; Th<sub>CO<sub>2</sub></sub>: CO<sub>2</sub> oil bath temperature; T<sub>CCO<sub>2</sub></sub>: CO<sub>2</sub> cooling bath temperature; P<sub>sat</sub>: saturation pressure

**Table S2**–Process yield and physicochemical characteristics of the SASD HPC powders

HPC (%w/v)	Yield, η (%)	D <sub>v,50</sub> (μm)	span	Moisture Content (%H <sub>2</sub> O/g <sub>powder</sub> )	a <sub>BET</sub> (m <sup>2</sup> /g)	SEM (shape/ surface)	XRPD (solid- state)
2.5	34	20.24	1.39	n.a.*	1.95	n.a.	Amorphous
5.0	66	19.95	1.44	4.3 ± 0.6	2.62	n.a.	Amorphous
7.5	49	18.26	1.52	4.6 ± 0.4	2.08	Irregular/ smooth	Amorphous
Raw	n.a.	n.a.	n.a.	2.5 ± 0.4	0.80	n.a.	Amorphous

\* Test not performed due to the insufficient powder amount required for the characterization; n.a. = not available.

**Table S3**–Parameter level values used in 3<sup>2</sup> full factorial design.

Level of factors used in the formulation				
Factor		-1	0	+1
A	C <sub>_solids</sub> (%w/v)	2.5	5.0	7.5
B	Ethanol (%v/v)	20	45	70

C<sub>\_solids</sub>: solid contents

Levels: low (-1), medium (0) and high (+1).

**Table S4**–Standard experiment order matrix.

Exp. No.	A	B
1	-1	-1
2	-1	0
3	-1	+1
4	0	-1
5	0	0
6	0	+1
7	+1	-1
8	+1	0
9	+1	+1

**Table S5**–DPPH radical scavenging (%) of RSV tests.

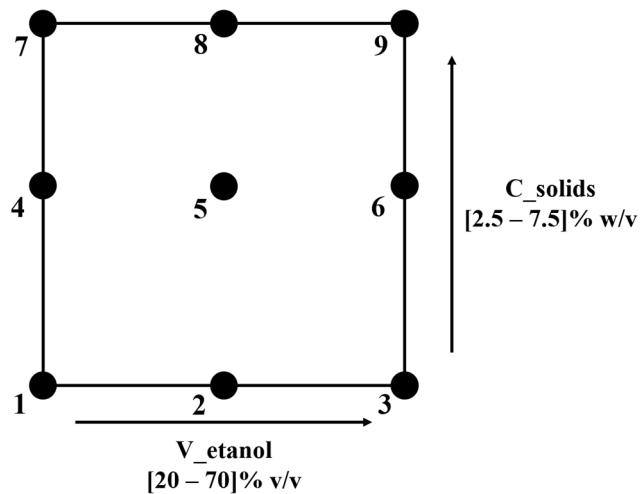
C <sub>RSV</sub> ( $\mu$ M)	raw RSV	RSV / 5.0 /		RSV / 7.5 / 70
		45	20	
100	10.75 $\pm$ 0.08	7.30 $\pm$ 0.32	11.54 $\pm$ 0.34	9.79 $\pm$ 0.31
400	28.40 $\pm$ 0.75	15.22 $\pm$ 0.75	19.32 $\pm$ 1.33	22.33 $\pm$ 0.57
800	43.01 $\pm$ 0.73	24.54 $\pm$ 0.67	33.60 $\pm$ 3.36	34.90 $\pm$ 0.34
1200	48.57 $\pm$ 5.63	38.63 $\pm$ 4.29	48.27 $\pm$ 0.33	43.86 $\pm$ 0.28
1500	53.75 $\pm$ 5.74	37.25 $\pm$ 7.21	52.26 $\pm$ 1.01	47.60 $\pm$ 0.35
1800	66.22 $\pm$ 2.50	46.87 $\pm$ 0.46	55.45 $\pm$ 0.70	43.49 $\pm$ 2.81
2100	62.24 $\pm$ 1.32	49.64 $\pm$ 0.63	58.09 $\pm$ 0.50	49.92 $\pm$ 0.74

**Table S6**–DPPH radical scavenging (%) of GA tests.

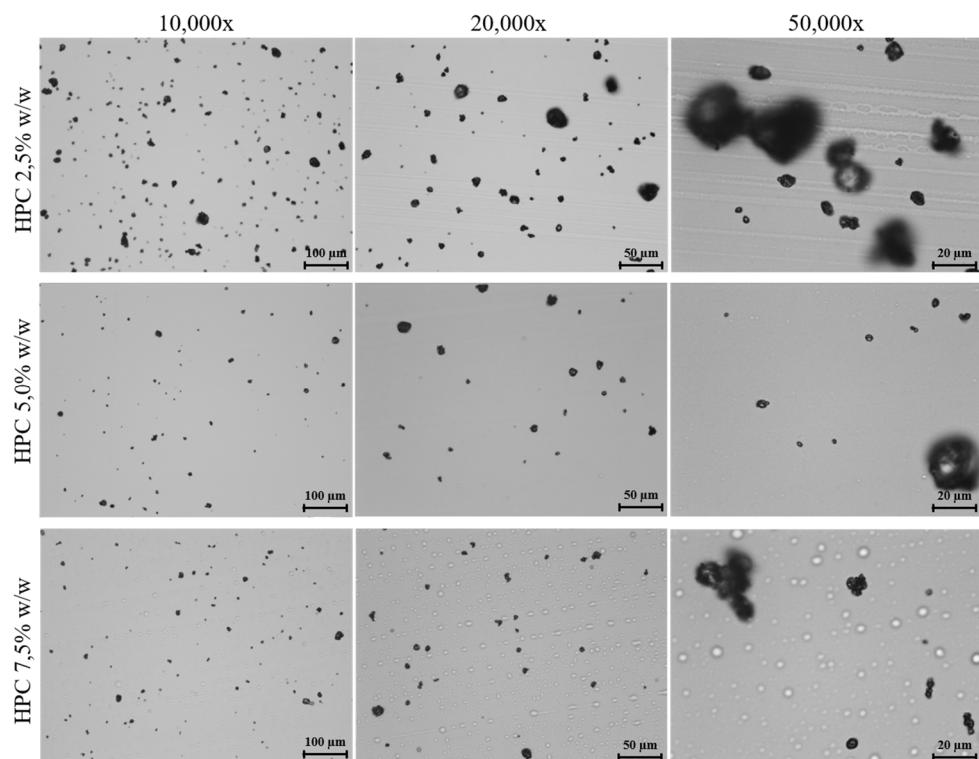
<b>C<sub>RSV</sub> (μM)</b>	<b>raw GA</b>	<b>GA / 5.0 / 45</b>	<b>GA / 7.5 / 20</b>	<b>GA / 7.5 / 70</b>
50	<b>11.31 ± 7.18</b>	<b>8.47 ± 1.54</b>	<b>15.18 ± 1.13</b>	<b>9.48 ± 4.58</b>
120	<b>31.50 ± 1.04</b>	<b>24.44 ± 7.98</b>	<b>41.67 ± 2.72</b>	<b>43.72 ± 1.83</b>
190	<b>60.67 ± 1.15</b>	<b>49.96 ± 8.20</b>	<b>75.95 ± 3.64</b>	<b>76.82 ± 4.25</b>
260	<b>79.00 ± 5.48</b>	<b>59.51 ± 13.75</b>	<b>82.97 ± 2.42</b>	<b>82.92 ± 1.25</b>
330	<b>88.54 ± 0.15</b>	<b>59.69 ± 14.34</b>	<b>85.46 ± 0.16</b>	<b>81.12 ± 3.55</b>
400	<b>89.17 ± 0.46</b>	<b>61.58 ± 10.72</b>	<b>75.47 ± 1.18</b>	<b>83.86 ± 0.52</b>
470	<b>90.00 ± 0.12</b>	<b>80.53 ± 3.46</b>	<b>76.10 ± 1.01</b>	<b>84.79 ± 1.25</b>

**Table S7**–Input and released amount of RSV and GA from the *in vitro* release tests.

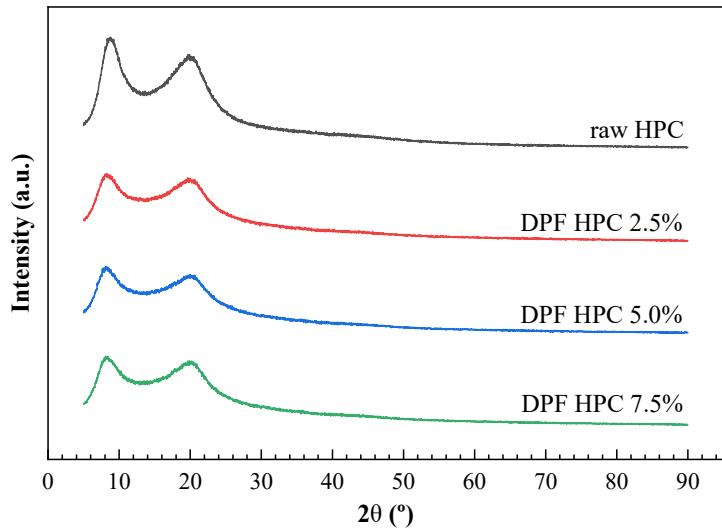
<b>Sample</b>	<b>C<sub>input</sub> (μg/mL)</b>	<b>μg phenolic input/ 100mg DPF</b>	<b>C<sub>máx</sub> (μg/mL)</b>	<b>μg phenolic rel./ 100mg DPF</b>	<b>%released</b>
Raw RSV	40.9 ± 0.2	–	1.4 ± 0.4	–	3 ± 1
DPF RSV/5.0/45	40.714 ± 0.008	1017	8 ± 1	201 ± 32	20 ± 3
Raw GA	41.7 ± 0.7	–	37 ± 2	–	92 ± 3
DPF GA/5.0/45	40.354 ± 0.006	877	37.2 ± 0.2	818 ± 6	93.4 ± 0.8



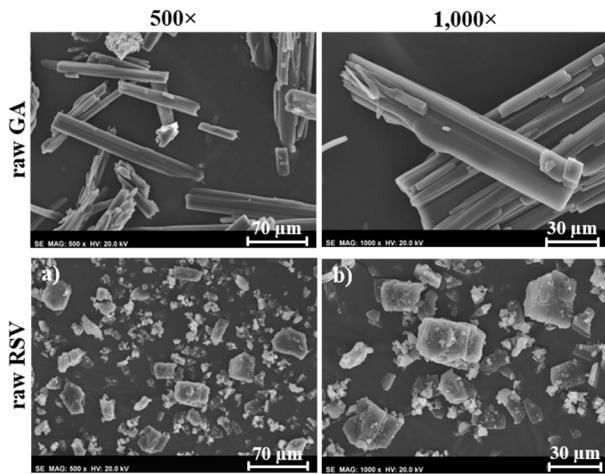
**Figure S1**—Schematic representation of the 3<sup>2</sup> full factorial DoE.



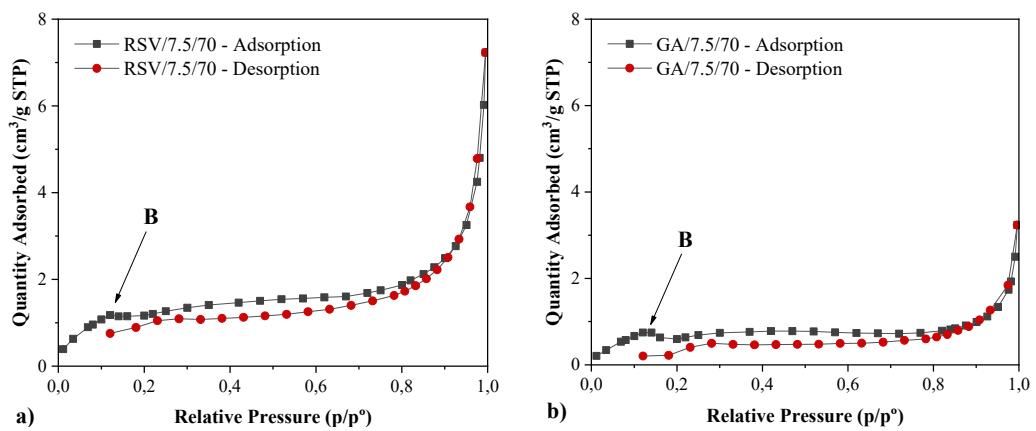
**Figure S2**—Morphologi G3 images of the DPFs microparticles with 2.5, 5.0 and 7.5 %w/v of HPC at different image magnifications: 10,000 $\times$ , 20,000 $\times$  and 50,000 $\times$ .



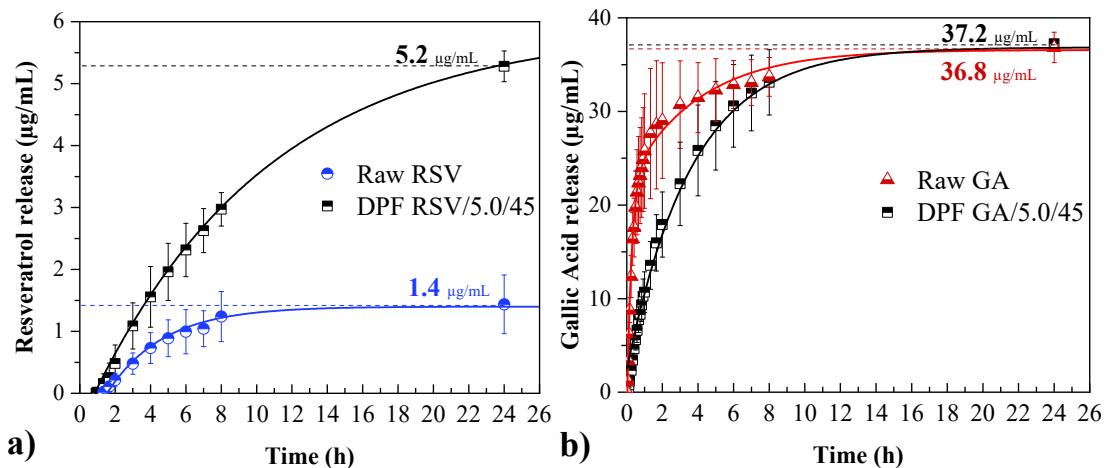
**Figure S3**–XRPD diffraction spectra of raw HPC powder and DPFs with 2.5, 5.0 and 7.5 %w/v HPC.



**Figure S4**–SEM images of raw GA and raw RSV at 500× and 1,000× magnifications.



**Figure S5**–Isotherm plots for a) HPC/RSV and b) HPC/GA microparticles obtained by SASD.



**Figure S6**–In vitro phenolic release profiles in pH 5.5 PBS at 32 °C for 24 hours from a) raw RSV and DPF RSV/5.0/45 samples and b) raw GA and DPF GA/5.0/45 samples, expressed in  $\mu\text{g}$  of phenolic per mL of solution.