Antibacterial Melt Blown PP - g – NDAM
Nonwoven

STAMP 2010
Sustainable Textiles and Medical Protection

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Introduction

- **Face Mask classified by use:**
  - Anti-dust face mask
  - Medical face mask
  - Anti-oil face mask

- **Face Mask Annual Output in China: 30 billions**

Distribution of main face mask products
Introduction

- **Medical Face Mask Classified by Structure**
  - Plane Mask
  - Molded Mask

Fig. 1 Molded Mask Structure
Introduction

**Materials of Molded Medical Face Mask**

- **4 layers**
  - **Outer layer: Spun-bond**
    - Fibers fineness: 16~20 μm
    - Composition: PP
  - **Interface layer: Melt Blowing**
    - Fibers fineness: 1.5~2 μm
    - Composition: PP
  - **Skeleton layer: Needle Punching**
    - Fibers fineness: 14~25 μm
    - Composition: PET
  - **Inner layer: Spun-bond**
    - Fibers fineness: 16~20 μm
    - Composition: PP

![Spun-bond](image1)
![Melt blowing](image2)
![Needle Punching](image3)

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Introduction

• **Antibacterial Medical Face Mask**

◆ *Nano Sliver Antibacterial Medical Face Mask*
  ➢ Mechanism: Nano-silver damages the cell wall and genetic composition of bacteria
  ➢ Non-reusable
  ➢ Low Sterilizing Rate
  ➢ Short time use

◆ *Antibacterial PP-g-NDAM Medical Face Mask*
  ➢ Active chlorine kill bacteria
  ➢ Active chlorine can be rechargable
  ➢ More stable antibacterial property
  ➢ High Sterilizing Rate
  ➢ Long time use
Introduction

◆ Grafting Reaction of PP with NDAM
N- halamine precursor, 2,4-diamino-6-diallylamino-1,3,5-triazine (NDAM)

Scheme 2. Reaction of PP with NDAM in reactive extrusion.

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Melt Blown Process

Polymer Melt Blown Device

Raw materials
Feeding machine
Dehumidifier
Air heaters
Filter
Reducing valve
Air tank
Steam separator
Air compressor
Feeding funnel
Screw extrude
Buffer head
Raising platform
Separator
Die
Receiving device

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Melt Blown Forming

**Outline**

1\(^{st}\) experiment*:
- Melt blown Process
- Performance testing of raw materials
- Basic Property test
- SEM

2\(^{nd}\) & 3\(^{rd}\) experiment*:
- Melt blown process
- Basic Property test
- Fineness & Pore diameter test (for 3\(^{rd}\) sample)
- Filtration test (for 3\(^{rd}\) sample)
- SEM

Antibacterial test:
- Content of active chlorine test (for the 3\(^{rd}\) sample)
- Antibacterial property test (for the 3\(^{rd}\) sample)

* The PP-g-NDAM which used in experiment 1\(^{st}\), 2\(^{nd}\) & 3\(^{rd}\) were synthesized at different time.
Melt Blown Process

PP - g – NDMA Melt Blowing Process

1. Polymer melt
2. Hot air
3. Inlet region
4. Orifice flow region
5. Swelling region

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Melt Blown Process

**Characters of Melt Blown Materials**

- Superfine fibers: 1.5~2.5μm (Fig. 1)
- Randomized crossover arrangement of fibers
- Many curved channels (Fig. 2)
- Small pore diameter (Fig. 3)
- Fluffy structure
Melt Blown Process

◆ **Parameters of Melt blown process**
  - Spinning temperature: 300 ~ 350 °C
  - PP MFI 25
  - Diameter of Orifice: 0.25mm
  - length to diameter ratio of Screw Extruder: 35:1

◆ **Problems during Melt blown Process**
  - Easy to high-temperature degradation of NDAM
  - Pungent odor of the fabrics
  - Fabric are yellow
  - Orifices blockage
  - Filament breakage
  - Shots in the fabric
The 1st Experiment

Basic Performance

<table>
<thead>
<tr>
<th>Content of modified pp(%)</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>40</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (mm)</td>
<td>1.08</td>
<td>0.67</td>
<td>0.56</td>
<td>0.53</td>
</tr>
<tr>
<td>Weight (g/m²)</td>
<td>77.69</td>
<td>75.56</td>
<td>72.14</td>
<td>71.87</td>
</tr>
<tr>
<td>Tensile (N)</td>
<td>7.65</td>
<td>6.89</td>
<td>3.76</td>
<td>3.48</td>
</tr>
<tr>
<td>Elongation(%)</td>
<td>16.6</td>
<td>18.84</td>
<td>10.24</td>
<td>8.74</td>
</tr>
</tbody>
</table>

Tensile Strength (N)

Elongation %

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Melt Blown Process

The 1st Experiment

• Fabric Structure

10% modi PP

20% modi PP

30% modi PP

40% modi PP

50% modi PP

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Melt Blown Process

Parameters of Melt blown process

- Spinning temperature: 240 ~ 280°C
- PP MFI 1500
- Diameter of Orifice: 0.25mm
- Length to diameter ratio of Screw Extruder: 35:1

Melt blown Process

- The 2\textsuperscript{nd} experiment is better than the 1\textsuperscript{st}
- The 3\textsuperscript{rd} experiment is the best
  - Easy to forming
  - White fabric
  - Feel fluffy and smooth of the fabric
  - Non-pungent odor of the fabrics

The 2\textsuperscript{nd} & 3\textsuperscript{rd} Experiment
Melt Blown Process

• Fabric Structure

The 2nd experiment

The 3rd experiment

30% PP- g - DAM

40% PP- g - DAM

60% PP- g - DAM

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Melt Blown Process

- **Fibers Fineness**

<table>
<thead>
<tr>
<th>Content of PP-g-NDAM (%)</th>
<th>30</th>
<th>40</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fibers Fineness (μm)</td>
<td>14.75±10.06</td>
<td>3.61±1.46</td>
<td>4.1±1.82</td>
</tr>
</tbody>
</table>

**The 2nd & 3rd Experiment**

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## Melt Blown Process

### Basic Performance

<table>
<thead>
<tr>
<th>Content of PP-g-NDAM (%)</th>
<th>Thickness (mm)</th>
<th>Weight (g/m²)</th>
<th>Tensile (N)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>0.91</td>
<td>44.47</td>
<td>8.5406</td>
<td>76.82</td>
</tr>
<tr>
<td>60</td>
<td>0.74</td>
<td>37.79</td>
<td>6.8974</td>
<td>73.88</td>
</tr>
</tbody>
</table>

The 2nd & 3rd Experiment

![Tensile strength curve diagram](image)

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**Melt Blown Process**

*The 2nd & 3rd Experiment*

**Pore Size Test**

Instrumentation: Capillary Flow Porometer

<table>
<thead>
<tr>
<th>Content of PP-g-NDAM (%)</th>
<th>Thickness (mm)</th>
<th>Mean Flow Pore Diameter (µm)</th>
<th>Bubble Point Pore Diameter (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>0.91</td>
<td>8.70</td>
<td>20.52</td>
</tr>
<tr>
<td>60</td>
<td>0.74</td>
<td>7.79</td>
<td>19.28</td>
</tr>
</tbody>
</table>

Pore Size Test

Instrumentation: Capillary Flow Porometer

40% PP-g-NDAM

60% PP-g-NDAM
Melt Blown Process

◆ Filtration Efficiency Test
  • Instrumentation: TSI8130
  • Particulate matter: Nacl
  • Particle diameter: 0.26um

<table>
<thead>
<tr>
<th>Sample</th>
<th>Flow (L/min)</th>
<th>Respiratory resistance (mmH₂O)</th>
<th>Filtration efficiency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure PP</td>
<td>32</td>
<td>1.81</td>
<td>35.16</td>
</tr>
<tr>
<td>40% pp-g-NDAM</td>
<td>32</td>
<td>1.1</td>
<td>30</td>
</tr>
<tr>
<td>60%pp-g-NDAM</td>
<td>32.1</td>
<td>1.33</td>
<td>28.77</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample</th>
<th>Flow (L/min)</th>
<th>Respiratory resistance (mmH₂O)</th>
<th>Filtration efficiency after electret 30kv (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure PP</td>
<td>32</td>
<td>1.9</td>
<td>84.04</td>
</tr>
<tr>
<td>40% pp-g-NDAM</td>
<td>32</td>
<td>1.1</td>
<td>79.56</td>
</tr>
<tr>
<td>60% pp-g-NDAM</td>
<td>32.1</td>
<td>1.33</td>
<td>77.41</td>
</tr>
</tbody>
</table>
Melt Blown Process

- **Mechanism of filtration**
  - Colliding
  - Electrostatic adherence
  - Inertial impaction
  - Diffusion effect
  - Gravitational

**Factors affect filtration efficiency**
- Diameter of fibers
- Mean Flow Pore Diameter
- Electret
- Thickness

The 2nd & 3rd Experiment

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**Melt Blown Process**

- **Respiratory resistance**
  - Medical respiratory resistance $\leq 35\text{mmH}_2\text{O}$ (GB2626-2006)
  - Melt blown of Medical face mask respiratory resistance : $1\sim 6\ \text{mmH}_2\text{O}$

<table>
<thead>
<tr>
<th></th>
<th>Pure PP melt blown</th>
<th>Melt blown after nano coating</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistance</td>
<td>5.4 mmH$_2$O</td>
<td>151.9 mmH$_2$O</td>
</tr>
<tr>
<td>Filtration efficiency</td>
<td>99.92%</td>
<td>95.6%</td>
</tr>
</tbody>
</table>

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Evaluation of Antibacterial Properties

Mechanism of Antibacterial PP-g-NDAM

Scheme 3. Simplified mechanism of biocidal polypropylene.
Evaluation of Antibacterial Properties

Active chlorine (ppm)

<table>
<thead>
<tr>
<th></th>
<th>Extracted in acetone</th>
<th>Washed in acetone</th>
<th>Unwashed</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP-g-DAM meltblown</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60% PP-g-DAM</td>
<td>95.49</td>
<td>210.4</td>
<td>336.07</td>
</tr>
<tr>
<td>40% PP-g-DAM</td>
<td>87.65</td>
<td>185.04</td>
<td>209.32</td>
</tr>
</tbody>
</table>

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# Evaluation of Antibacterial Properties

## Antibacterial result of cleaned samples

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>S</th>
<th>Control</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>40%</strong></td>
<td>x10^0</td>
<td>x10^1</td>
<td>x10^2</td>
<td>x10^3</td>
</tr>
<tr>
<td><strong>S.Auraus</strong> (c=1x10^5)</td>
<td>8</td>
<td>86</td>
<td>10.5</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>65.5</td>
<td>6.5</td>
<td>0.5</td>
</tr>
<tr>
<td><strong>60%</strong></td>
<td>x10^0</td>
<td>x10^1</td>
<td>x10^2</td>
<td>x10^3</td>
</tr>
<tr>
<td><strong>Control</strong></td>
<td>8</td>
<td>105</td>
<td>12.5</td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>49.5</td>
<td>5</td>
<td>0.5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>S</th>
<th>Control</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>40%</strong></td>
<td>x10^0</td>
<td>x10^1</td>
<td>x10^2</td>
<td>x10^3</td>
</tr>
<tr>
<td><strong>E.Coli</strong> (c=3.0x10^5)</td>
<td>8</td>
<td>233.5</td>
<td>46</td>
<td>5.5</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>220.5</td>
<td>33.5</td>
<td>4</td>
</tr>
<tr>
<td><strong>60%</strong></td>
<td>x10^0</td>
<td>x10^1</td>
<td>x10^2</td>
<td>x10^3</td>
</tr>
<tr>
<td><strong>Control</strong></td>
<td>8</td>
<td>275</td>
<td>68.5</td>
<td>6.5</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>218</td>
<td>24.5</td>
<td>1.5</td>
</tr>
</tbody>
</table>

**Standard: AATCC100**
Conclusion

- The Melt-blown web forming technology of PP-g-NDAM is feasible.
- Key technology: control of fiber fineness distribution and fiber web evenness, besides, electret processing can increase filtration efficiency without increasing respiratory resistance.
- Anti-bacterial mask has a broad market prospect.
- Control the spinning temperature of melt blown process.
- Raw material affects the property of modified PP fabric.
- The best property: 60% PP-g-NDAM.
- The grafting ration affects the antibacterial.
Prospect

◆ **Optimize Melt Blown Technology**
  - Decrease the temperature of melt blown technology
  - Decrease Fiber fineness $\leq 2\mu m$
  - Increase Filtration of Melt Blown Materials $> 90$
  - Improve the mechanical properties

◆ **Antibacterial Properties**
  - Improve the grafting ration of PP and NDAM
  - Increase Antibacterial rate $\geq 99.9$

◆ **Realizing industrialization of face mask**
The End

Thank you!